SIMULATION OF BATCH DISTILLATION COLUMN

RONNIE TAN BOON HOW

**A project report submitted in partial fulfilment of the
requirements for the award of the degree of
Bachelor (Hons.) of Chemical Engineering**

**Faculty of Engineering and Science
Universiti Tunku Abdul Rahman**

September 2011

DECLARATION

I hereby declare that this project report is based on my original work except for citations and quotations which have been duly acknowledged. I also declare that it has not been previously and concurrently submitted for any other degree or award at UTAR or other institutions.

Signature : ________________________

Name : Ronnie Tan Boon How

ID No. : 08UEB01855

Date : 22nd September 2011

APPROVAL FOR SUBMISSION

I certify that this project report entitled **“SIMULATION OF BATCH DISTILLATION COLUMN”** was prepared by **RONNIE TAN BOON HOW** has met the required standard for submission in partial fulfilment of the requirements for the award of Bachelor of Engineering (Hons.) Chemical Engineering at Universiti Tunku Abdul Rahman

Approved by,

Signature : _________________________

Supervisor : Ms. Teoh Hui Chieh

Date : _________________________

ACKNOWLEDGEMENTS

I am heartily thankful to my supervisor in this project, Ms. Teoh Hui Chieh, for her endless guidance, advises, and her patience throughout this project.

I would like to express my greatest gratitude to my family member especially my sister and parent for their endless financial support throughout my four years stint of studies in UTAR.

Thanks to my course mates who has helped me out on this project. Their encouragement and helpfulness is much appreciated and never would I forget.

Last but not least, to my girlfriend Siang Ling, thank you for your support, understanding and love

SIMULATION OF BATCH DISTILLATION COLUMN

ABSTRACT

Batch distillation has regained it interest from industries recent years. This is mainly due to its flexibility and capability to produce high purity product. This interest has drawn many researcher attentions to invent a fast and reliable model to serve the industry. To date, several type of simulation model had been presented, and the interest to obtain a better control and improvement on batch distillation has yet to stop. In this project, simulation of binary mixture batch distillation was done by using graphical method and computer simulator, Aspen Batch Distillation (ABD). The results from these simulations were used as a basis to validate the results obtained (by others) experimentally from an existing column in Universiti Tunku Abdul Rahman, Unit Operation Laboratory, which is suspected to be faulty. Comparison between the compositions obtained from graphical method with ABD shows only slight error. It has a maximum error of 5.2 %. With this minor error, the two methods supported each other and show confident in the results obtained. The experiment results, however, gives an enormous error of over 175 % compared to ABD. Responding to this large error, troubleshooting was carried out on the existing column and a faulty reflux splitter was identified. The splitter was not functioning properly causing incorrect split of reflux and distillate. Another reason for the inaccurate results is due to the method in measuring the composition of the samples. The compositions obtained from the experiment were determined using Refractive Index method which has a strong function of temperature. This temperature dependency was suspected to cause error in the experimental results. It is suggested that a more accurate method such as back titration method and gas chromatography to be used for determination of composition.

TABLE OF CONTENTS

CHAPTER

LIST OF TABLES

LIST OF FIGURES

LIST OF SYMBOLS / ABBREVIATIONS

W	molar holdup at bottoms, kmol
W_0	initial molar holdup at bottoms, kmol
W_t	molar holdup at bottom at any time *t*, kmol
W_k	molar holdup at bottoms at step *k*, kmol
W_{k-1}	molar holdup at bottoms at step *k -1*, kmol
x	mole fraction of component
x_{W_0}	initial bottoms mole fraction of lighter component at
x_{W_t}	bottoms mole fraction of lighter component at any time *t*
x_{W_k}	bottoms mole fraction of lighter component at step *k*
$x_{W_{k-1}}$	bottoms mole fraction of lighter component at step *k-1*
x_{D_t}	distillate mole fraction of lighter component at any time *t*
x_{D_t}	distillate mole fraction of lighter component at any time *t*
$x_{D,avg}$	average distillate mole fraction of lighter component at any time *t*
x_{D_k}	distillate mole fraction of lighter component at step *k*

V	boilup rate, kmol/s
T	temperature, K
T_c	critical temperature, K
Q	heat input, kW
H	enthalpy of vaporization, kJ/mol
D	distillate, kmol
R	reflux ratio
R_k	reflux ratio at step *k*
t	time, s
A, n	regression of chemical compound

ABD	Aspen Batch Distillation
SD	standard deviation
UTAR	Universtiti Tunku Abdul Rahman
FES	Faculty of Engineering and Sciences

LIST OF APPENDICES

CHAPTER 1

INTRODUCTION

1.1 Background

Distillation is the most commonly used approach for separation of homogeneous fluid mixture. Separation is done by taking advantage of boiling point different or volatility between the components in the mixture; by preferentially boiling the more volatility component out of the mixture (Smith & Jobson, 2000). When a mixture of liquid of two volatile liquid is heated, the vapour that comes off will contained higher concentration of the more volatile material as compared to the liquid in the reboiler. On contrary, when the vapour cooled the higher-boiling point material have better tendency to condense than the more lower-boiling-point material (Kister, 1992).

Distillation is arguably as one of the oldest separation/purifying liquid mixture technology (Mujtaba, 2004). This ancient unit operation has been practiced for over hundreds of years ago mainly for alcoholic beverage purifying. This operation has expanded from a way to enhancing alcoholic beverage to a prime separation, purification and waste removal process in chemical industries at beginning on the twentieth century. Today, distillation is so widely used as compare to other separation techniques in spite extreme high usage of energy due to distillation can handle extremely large and extremely low flowrate, its ability to separate feed with wide range of feed concentration and ability to produce high purity (Smith & Jobson, 2000), and according to Kister (1992), the superiority of distillation for separation of fluid mixture is a fundamental, and hence, it is unlikely to be displaced.

Distillation process can divided into two classes; batch distillation and continuous distillation. Batch distillation is especially important in fine specialty, pharmaceutical, essential oil and some petroleum product. In a conventional batch distillation column feed will be initially charged into the reboiler and nothing is added to the process till end. The key different between this two distillation processes is that, feed will be constantly supplied to continuous column and making it a steady state operation. As for batch distillation, the composition of higher boiling point component will gradually concentrated over time, making it a time varying process.

Even though continuous distillation has outshine batch distillation process since 1950, Rippin (1983) reported that it is astonishing to find that "large proportion of world‘s chemical production by volume and a much larger proportion by value still made in batch plants and it does not seem likely that this proportion will decline." This statement clearly shown that the trend of batch distillation still has it value and Biegler et al. (1997) claimed "with recent trend of building small flexible plants that are close to market consumption, there has been renewed interest in batch process." Due to flexibility of batch process, batch distillation is preferable compared to continuous distillation when high-value-added, low volume chemical must be separated. It is also important for material dealt in irregularly or seasonally scheduled period. With this flexibility, batch distillation able to coup with uncertainties in feed and product specification. Furthermore, several type of mixture can be handled by switching the operation condition of the column (Kim & Diwekar, 2005). Another main advantage of batch distillation to continuous distillation lies in the use of single column. For a multi-components liquid with n_c number of components generally required (n_c-1) numbers of continuous-column for separation. For batch distillation, however, only one column and a sequence is needed (Mujtaba, 2004). Pharmaceutical industry and speciality chemical industry are example of industries that uses batch distillation in their production line (Kaama, Rodríguez-Donisa, & Gerbaud, 2008).

Current technology has made simulation of a batch distillation, or other batch processes, much easier and less complicated compared to years back. Simulation is usually the first step to conduct feasibility study of a design before it can be implement into a pilot plant or industrial scale plant. Not only feasibility study, simulation give a better understanding of a process, approximation of the cost to setup and potential challenges that the process may face upon setting up the design to industrial scale.

Simulation of a batch distillation column can be done by using mathematical model graphical method, shortcut method and rigorous method (Kim & Diwekar, 2005; Seader & Ernest, 2006), and computer simulator such as Aspen Batch Distillation (ABD), BATCH-DIST and Batchsim.

1.2 Problem Statement

An existing batch distillation column in Universiti Tunku Abdul Rahman, Unit Operation Laboratory is suspected to be faulty. In order to verify this issue, simulation of this batch distillation column is to be performed by using computer aided design software and mathematical model so that the results can be compared and validated.

1.3 Aims and Objectives

Simulation on batch distillation column is done by using graphical method and Aspen Batch Distillation (ABD) simulator to examine the treand of distillate and bottoms composition at constant reflux and regulating reflux. Both of the results from the simulation will be compared to the results obtained experimentally (by others) using an existing column in Universiti Tunku Abdul Rahman Unit Operation Laboratory. Thus, experimental results can be validated.

CHAPTER 2

LITERATURE REVIEW

2.1 Background

The earliest form of batch distillation, also known as Rayleigh distillation, was presented in by Lord Rayleigh (1902). In his model, there is only a column, a condenser and a still pot. No reflux is returned to the still and no stage or packing material inside the column. Hence, Rayleigh distillation is simply a one stage distillation, and the analysis is based on material and component balance (Kim & Diwekar, 2005). Figure 2.1 shows a conventional batch distillation column with N number of stages, bottoms holdup, distillate and heat supply.

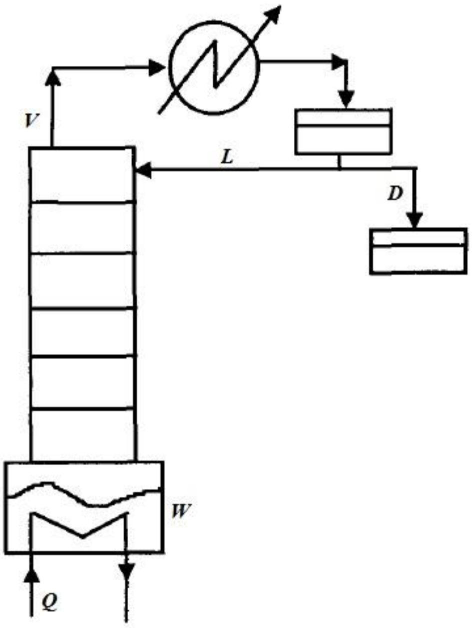


Figure 2.1: Conventional Batch Distillation Column

Batch distillation column can be operated with three reflux policies in order to meet certain product specification. The reflux policies that can be employed to a batch distillation are either constant reflux, regulating reflux or optimum reflux. For constant reflux, distillate purity for lighter component will drop over time. In order to maintained the purity of distillate over time, regulating reflux policy can be employed. Optimum reflux policy is used when maximum profit from the operation is required; it is a trade off policy between constant reflux and regulating reflux policies (Kim & Diwekar, 2005).

Batch distillation column can also be operated in various configuration. Figure 2.2 below shows arrangement of how batch distillation can be configured. Configuration (a) in Figure 2.2 is the most common type of configuration that batch distillation column is operated in laboratory, with reboiler at the bottom and condensor at the top (Kim & Diwekar, 2005).

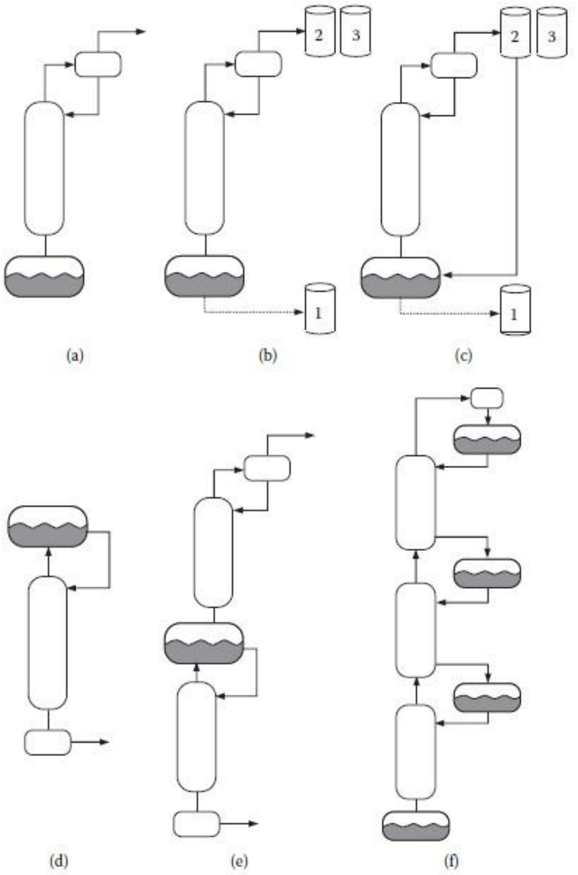


Figure 2.2: Configuration of Batch Distillation Column

2.2 Development of Batch Distillation

Bogart (1937) is the first to analyse of variable reflux policy for binary batch distillation. His method was based on simplified Rayleigh Equation and maintains the distillate composition throughout the process. The method presented by Bogart was using McCabe-Thiele Diagram to determine a set of various values of lighter component at bottoms, x_w at a fixed value of distillate composition, x_D with different gradient of operating line. The slope of the gradient can be represent by $\frac{R}{1+R}$, where R is the reflux ratio. The procedure proposed was based on trial and error. In his analysis, the model is assumed to have negligible column holdup and constant molar overflow (Seader & Ernest, 2006; Kim & Diwekar, 2005).

Smoker and Rose (1940) presented the first analysis of constant reflux binary batch distillation column. In their model, it was assumed that there is no column holdup. Smoker and Rose used Rayleigh's Equation together with McCabe-Thiele diagram to capture the dynamic of a batch distillation column. In their method, the relationship of x_W and x_D is repeatedly determined by using McCabe-Thiele diagram. With the obtained relationship, Rayleigh equation can be solve by graphically integrate $1/(x_D - x_W)$ versus x_W and the area under the curve between the feed composition and bottoms composition can be find to obtain the value of integral. Their method, however, is independent from time (Seader & Ernest, 2006; Kim & Diwekar, 2005).

Meadow (1963) developed the first comprehensive model of a multicomponent batch distillation. In his model, he included equation for material, energy and volume balance around theoretical trays. The model is assumed to have constant-volume tray holdup, negligible vapour holdup, perfect mixing on every trays and adiabatic operation. However, no experimental verification of the model was presented (Ugur, 2002).

A mathematical model of multicomponent batch distillation was presented by Distefano (1968). He gave a step by step calculation of the algorithm of the model. The study mainly focuses on analysis of the most widely used numerical integration routine used in simulation model. The mathematical model was derived from material and energy balances around different stages of the column and incorporated the same assumption made by Meadows (1963). Distefano (1968) added, due to radical changes in composition on each tray, the assumption of constant-mass tray holdup was unacceptable. Due to this reason, Distefano (1968) made assumption of constant volume holdup instead. His works is now used as a basis for almost all of rigorous modelling of batch distillation (Ugur, 2002).

Diwekar and Madhavan (1991) proposed a short-cut method to relax some of the problem faced by rigorous model presented by Meadows (1963) and Distefano (1968). According to them, the rigorous model simulation of batch distillation required large computational time and huge memory requirement, and are too complex to be used in optimal design calculation. The methods presented by them are based on modified version of Fenske-Underwood-Gilliland method which is used for continuous distillation column. The technique was applied to both binary and multicomponent distillation under constant reflux and variable reflux. Their results was then compared to plate-to-plate calculation of rigorous model and the results predicted from the short-cut model matched quite well with the results of rigorous model (Diwekar & Madhavan, 1991). Additional, increasing of stages in the column will have no effect on computational time of this short-cut model.

Sundaran and Evans (1993) did a work on constant reflux as well. In the paper presented, they used the Fenske-Underwood-Gilliland method for continuous distillation method directly and developed a time explicit model. The different of their model comparing to Diwekar and Madhavan (1991) is that the model they developed was based on time as an independent variable, while Diwekar and Madhavan (1991) used the bottoms composition as independent variable.

Bonsfills and Puigjaner (2004) worked out a simplified rigorous mathematical model based on mass balance, vapour-liquid equilibrium equation and constant reflux ratio. The simulated results was extensively validated experimentally, and compared with results from commercial computer software simulator. Their simulation works was the first to used both constant and variable relative volatility. With the aids from Wilson's equation, both the constant and variable relative volatility can be calculated. In their finding, for methanol-water mixture, binary batch distillation was best to be simulated by constant relative volatility if the reflux ratio is high. On the other hand, for low reflux ratio, say one, it was reported that by using variable relative volatility will give the most satisfactory results comparing to experimental results. This simplified rigorous method was also tested for ternary and binary azeotropic mixture. The results were satisfactory as well. Bonsfills and Puigjaner (2004) also did measure the temperature profile for some of the tray at their pilot plant experiment. The purpose was to check the concentration profile, even though it is not reliable.

2.3 Computer Aided Design for Batch Distillation

Kim and Diwekar (2005) mentioned the difficulties to analyse a batch distillation without assistant from computer-aided design software. The reason is because batch distillation is time varying process and involved complex numerical integration. To obtain a transients result, one has to opt different simulation models. Hence, to save time, computer aided simulation is becoming important to serve the current industry.

Kim and Diwekar (2005) stated good batch distillation software should consist of multiple models, options of reactive distillation and three phase distillation, and various configuration like semibatch, recycle waste cut and middle vessel column. Review of few batch distillation software; BatchSim, BatchFrac and MultiBatchDS, is given by Kim and Diwekar (2005) and Diwekar (1996). BatchFrac is later rename to BatchSep in year 2004 and again it rename to Aspen Batch Distillation in year 2008 as a component is AspenOne V7 for process engineering (Cook, Engel, & Zehnder, 2004; AspenTech, 2008)

CHAPTER 3

METHODOLOGY

3.1 Computer Software Simulation

Computer software simulation was done by using Aspentech Aspen Batch Distillation on the existing batch distillation column located in UTAR, FES, Unit Operation Laboratory. This column consists of eight trays, a reboiler and a condenser. The configuration of this column is a conventional type as shown in Figure 2.2 (a).

The simulation setting used for constant reflux policy was per existing column. The initial feed is ten litres made up of 25 v/v % ethanol-water mixture. Simulation time was forty minutes at constant reflux of 80 % and the result of distillate composition was obtained at interval of ten minutes.

The second of simulation was done by using regulating reflux policy. The initial used for simulation was the same as constant reflux policy. In this setting, simulation was carried out with a reflux of 40 %. In interval of ten minutes, reflux was increased by 10 % increment and result was obtained. The total time of simulation was forty minutes.

3.2 Mathematical Model Simulation for Binary Distillation

Before simulation can be done, the boil-up rate of the batch distillation operation needs to be calculated. This can be achieved by predicting the latent heat of vaporisation of the component involved at certain temperature by using modified Watson's equation (Coker, 2007; Martin & Edwards, 1964)

$$H = A\left(1-\frac{T}{T_c}\right)^n \tag{3.1}$$

where,
H = enthalpy of vaporisation, kJ/mol
T_c = critical temperature, K
T = temperature , K
A and n = regression coefficient for chemical compound

Once the latent heat of vaporisation was found, and together with the heat input to the system, the boil-up rate of the system can be calculated by using

$$Q = (x_e H_e + x_w H_w)V \tag{3.2}$$

where,
Q = heat input, kW
x_e = mole fraction of ethanol
x_w = mole fraction of water
H_e= enthalpy of vaporisation of ethanol, kJ/kmol
H_w= enthalpy of vaporisation of water, kJ/kmol
V = boilup rate, kmol/s

3.2.1 Distillation with Constant Reflux

Simulation of binary distillation with constant reflux ration can be done by using Rayleigh equation in conjunction with McCabe-Thiele graphical method (Kim &

Diwekar, 2005). Batch distillation under constant reflux can be analysed mathematically by considering that the mole lost from the still represent moles of distillate collected. Thus,

$$-dW=dD \tag{3.3}$$

The differential material balance for lighter component can be written as:

$$x_D dW = d(W x_w) = x_w dW + W dx_w \tag{3.4}$$

Rearranging and integrating,

$$\int_{W_0}^{W_t} \frac{dW}{W_t} = \ln\left(\frac{W_t}{W_0}\right) = \int_{x_{W_0}}^{x_{W_t}} \frac{dx_W}{x_D - x_W} \tag{3.5}$$

where,

x_{w_t}= bottoms composition of lighter component at any time, *t*

x_{w_0}= initial bottoms composition of lighter component

W_t= total holdup at bottoms at any time, *t,* kmol

W_0= initial charge, kmol

Equation (3.5) is simply known as Rayleigh equation. The bottoms holdup at any time time, *t* can be found by

$$W_t = W_0 - \frac{Vt}{R+1} \tag{3.6}$$

Where

W_t= total holdup at bottoms at any time *t,* kmol

W_0= initial charge, kmol

V= boilup rate, kmol/s

R= reflux ratio

By using the method proposed by Smoker and Rose (1940), the relationship of x_W and x_D can be recursively determined by McCabe-Thiele Diagram; by constructing a series of operating line of slope $\frac{R}{(R+1)}$ by trial and error and *N* numbers of stages are stepped off. The right hand side of the Rayleigh equation can be integrated by plotting $\frac{1}{x_D - x_W}$ vs x_W. The area under the curve between the initial composition of ethanol at bottoms, x_{W_0} and bottoms composition of ethanol at any time, x_{W_t} will give the value of integral which is $ln\frac{W_t}{W_0}$. This method, however, is assumed that no holdup in the tray and condenser. (Kim & Diwekar, 2005; Seader & Ernest, 2006; Kister, 1992). The average composition of distillate at any time *t* can be obtained by:

$$x_{D,avg}=\frac{W_0x_0\text{-}\ W_tx_{W_t}}{W_0\text{-}W_t} \tag{3.7}$$

where,

$x_{\mathrm{D,avg}}$= average composition of lighter component at distillate

3.2.2 Distillation with Regulating Reflux

Simulation of regulating reflux policy will be done by modifying Smoker and Rose method describe in section 3.2.1. First, Equation 3.3 will be modified to

$$\int_{W_{k-1}}^{W_k}\frac{dW}{W}=ln\left(\frac{W_k}{W_{k-1}}\right)=\int_{x_{W_{k-1}}}^{x_{W_k}}\frac{dx_W}{x_D\text{-}x_W} \tag{3.8}$$

where,

W_k = total holdup at bottoms at step *k*, kmol $\quad k = 1, 2, 3……$

W_{k-1} = total holdup at bottoms at step *k - 1*, kmol $\quad k = 1, 2, 3……$

X_{W_k} = bottoms composition of ethanol at step *k*, $\quad k = 1, 2, 3……$

$X_{W_{k-1}}$= bottoms composition of ethanol at step *k -1*, $\quad k = 1, 2, 3……$

Average composition of distillate collected for step *k* will be

$$x_{D_k} = \frac{W_{k-1}x_{k-1} - W_k x_{W_k}}{W_{k-1} - W_k} \tag{3.9}$$

and the cumulative distillate composition from beginning can be calculated by using

$$x_{D,avg} = \frac{\sum_0^k D_k x_{D_k}}{\sum_0^k D_k} \tag{3.10}$$

where,

D_k= total distillate collected at step *k* $k = 1, 2, 3\ldots\ldots$

The second step is to establish the relationship between *R*, x_D and x_W using McCabe-Thiele graphical method. Once the values of *R* are selected, the relationship between x_D and x_W will be obtained using methods described in section 3.2.1. Then the right hand side of the Rayleigh equation can be integrated by plotting $\frac{1}{x_D - x_W}$ vs x_W. The area under the curve between the initial bottoms compositions $x_{W_{k-1}}$ and the bottoms composition x_{W_k} at step *k* now give the value of integral which is $ln\frac{W_k}{W_{k-1}}$. Again, this modified method is based on assumption that no holdup in the tray and condenser. While distillation is proceeding, and the vapour boil *V* is fixed, the instantaneous distillate will vary according to the reflux ratio, which can be express as

$$\frac{dD}{dt} = V(1\text{-}L/V) \tag{3.11}$$

or

$$\frac{dD}{dt} = V\left(\frac{1}{R+1}\right) \tag{3.12}$$

and substituting equation 3.11 into equation 3.3, rearranging and integrating, holdup at the bottoms at step *k* can be calculated by

$$W_k = W_{k-1} - \frac{Vt}{R_k + 1} \tag{3.13}$$

where,

W_k = total holdup at bottoms at step k, kmol

W_{k-1} = total holdup at bottoms at step $k-1$, kmol

V= boil-up rate, kmol/s

R_k= reflux ratio at step k

3.3 Experimental Results Done by Others

The experimental results used in this project were obtained from previous experiment done by others.

3.3.1 Constant Reflux Percentage

The experiment starts up by vacuuming the column for at least ten minutes. The experiment was started with ten litres of 25 v/v % of ethanol-water mixture as initial feed to the reboiler. The initial refractive index of the feed is measured. The purpose of measuring the refractive index is to determine the composition of samples collected. The column is then allowed to run at total reflux for at least ten minutes after the top product appear at the reflux drum. The process is to stabilise the column into steady state. The reflux will then be lower to 80 % and the sample of top product was collected for reflective index measurement to obtained to determine the composition at interval of ten minutes. The process is allowed to run for forty minutes. At the end of the experiment, the total volume of the distillate will be measure. The result obtained will be tabulated and a graph of product composition versus time will be plotted. Figure 3.1 shows the configuration of column used in experiment

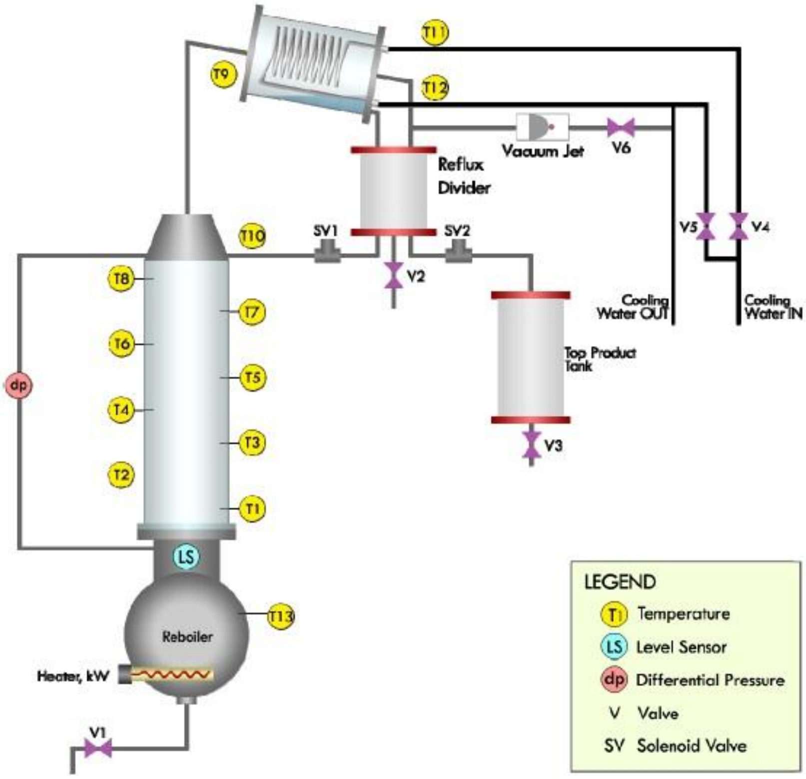


Figure 3.1: Configuration of Batch Distillation Column in UTAR, FES Unit Operation Laboratory

3.3.2 Regulating Reflux Percentage

This experiment will use the same feed and start up procedures as the experiment in Section 3.3.1. The experiment, however, will be carried out by increasing the reflux at the interval of ten minutes. Before regulating the reflux, a sample of top product will be taken for reflective index measurement to determine composition. The experiment is allowed to run for forty minutes. At the end of the experiment, the volume of the top product is measured. The result is then tabulate and a graph of average composition versus time is plotted.

CHAPTER 4

RESULTS AND DISCUSSIONS

4.1 Determination of Boil-up Rate

The boil-up rate, V, was estimated by using Equation (3.1) and Equation (3.2). The critical temperature, T_{c}, and regression coefficient A and n for ethanol and water given by Coker (2007) is as per Table 4.1.

Table 4.1: Critical Temperature T_c and Regression Coefficient A and n for Ethanol and Water

Compound	A	n	T_c (K)
Ethanol	43.122	0.079	516.25
Water	52.053	0.321	647.13

The initial condition of ten litres of 25 v/v% of ethanol in water was converted to mole fraction first

7.5 litres of water

7.5×10^{-3} m^3 $\times$ 1000 kg/m^3 $\div$ 18.02 kmol/kg = 0.4163 kmol

2.5 litres of ethanol

2.5×10^{-3} m^3 $\times$ 789 kg/m^3 $\div$ 46.07 kmol/kg = 0.0428 kmol

Total initial holdup, $W_{0,}$ = 0.4163 + 0.0428 = 0.4591 kmol

Mole fraction of ethanol

$$x_e = \frac{0.0428}{0.0428 + 0.4163}$$

$$x_e = 0.0933$$

Mole fraction of water

$$x_w = 1 - x_e$$

$$x_w = 0.9067$$

Using heat input of 1.85 kW and temperature at 85 °C, the boil-up rate, V, is found to be

$$H_e = A\left(1-\frac{T}{T_c}\right)^n$$

$$= 43.122\left(1-\frac{85 + 273.15}{516.25}\right)^{0.079}$$

H_e= 39.27341 kJ/mol or 39273.41 kJ/ kmol

and

$$H_w = A\left(1-\frac{T}{T_c}\right)^n$$

$$= 52.053\left(1-\frac{85 + 273.15}{647.13}\right)^{0.321}$$

H_w= 40.18427 kJ/ mol or 40184.27 kJ/kmol

$$Q = (x_e H_e + x_w H_w)V$$

$$V = \frac{Q}{(x_e H_e + x_w H_w)}$$

$$= \frac{1.85}{(0.093257)(39273.41)+(0.906743)(40184.27)}$$

V = 4.6135 × 10^{-5}kmol/s or 2.7681 ×10^{-3} kmol/min

In the experiment, the temperature at the pot is always maintained at 85°C. However, in calculation, it is impossible to predict the boil-up rate with constant heat transfer at a fixed temperature (Greves, Mujtaba, & Hussain, 2001). The argument behind this phenomenon is, the lighter component, which has lower heat of vaporisation, will gradually deplete and leaving the heavier component in the column. Hence, in order to maintain the boil-up rate, the heat input to the column has to be gradually increased. To reduce the complexity of the simulation model, the boil-up is assumed to be constant throughout the distillation.

4.2 Batch Distillation at Constant Reflux

4.2.1 Results From Graphical Method

With a known boil-up rate, simulation by using graphical method was done to find the composition at distillate and bottoms at any time, *t*. However, holdup at bottoms at any time *t,* need to be calculated first. Using time equal to ten minutes as example, Holdup at the bottoms W_{10} can be calculated by substituting the value of V into Equation (3.6). Reflux 80 % is equal to reflux ratio of four. This can be calculated by $\mathrm{R} = \frac{\text{reflux \%}}{\text{100 - reflux \%}}$

$$W_t = W_0 - \frac{Vt}{R+1}$$

$$W_{10} = 0.45913 - \frac{(2.7681 \times 10^{-3})(10)}{4+1}$$

W_{10}=0.45359 kmol

Then, the relationship of bottoms composition, x_W and distillate composition, x_D is recursively determined by drawing operating line of slope $\frac{R}{(R+1)}$, which is 0.8 at reflux 80 % at any point on the 45° line, and followed by eight stepping. The reason 8 stepping was drawn because the column used in the experiment consists of eight stages. Figure 4.1 shows an example of the McCabe-Thiele diagram and stepping for x_D (0.82, 0.82) that give x_W 0.195.

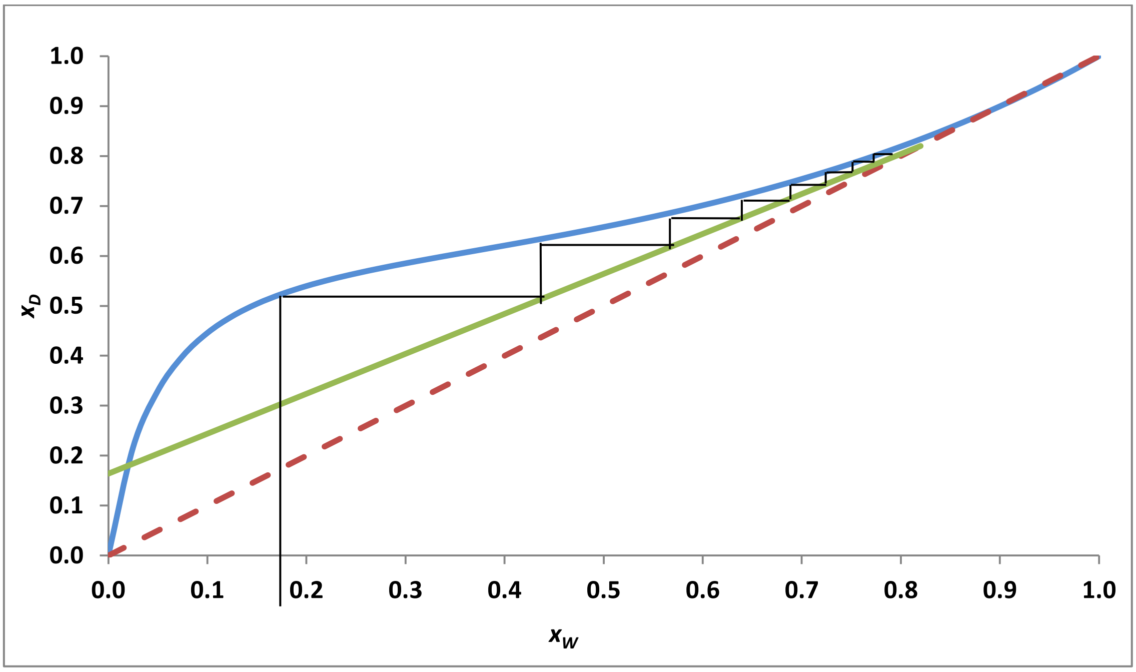


Figure 4.1: McCabe Thiele Diagram and Stepping from x_D (0.82, 0.82)

The relationship between x_D *and* x_W obtained from the method above is tabulated in Table 4.2 and a graph of 1/ x_D - x_W against x_W is plotted (Figure 4.2).

Table 4.2: Relationship Between x_D and x_W Obtained from McCabe-Thiele

x_D	x_W	$1/ (x_D-x_W)$
0.80	0.0250	1.290323
0.81	0.0850	1.379310
0.82	0.1950	1.600000

By using the initial condition and t = 10 minutes as example, the initial total holdup at the bottoms that calculated in Section 4.1 is 0.4591 kmol with ethanol and water mole fraction at 0.0933 and 0.9067 respectively. Value of x_D and x_{W_t} was calculated by using Equation (3.5) and Equation (3.7). As mentioned in Section 3.2.1, the area under the curve will give value of $ln\left(\frac{W_t}{W_o}\right)$, and to avoid graphical integration, an equation to fit the curve was obtained by using Microsoft Excel as shown in Figure 4.2. Hence, by solving the integration, the value of x_{W_t} was obtained. Below is the sample of calculation at the tenth minutes of distillation.

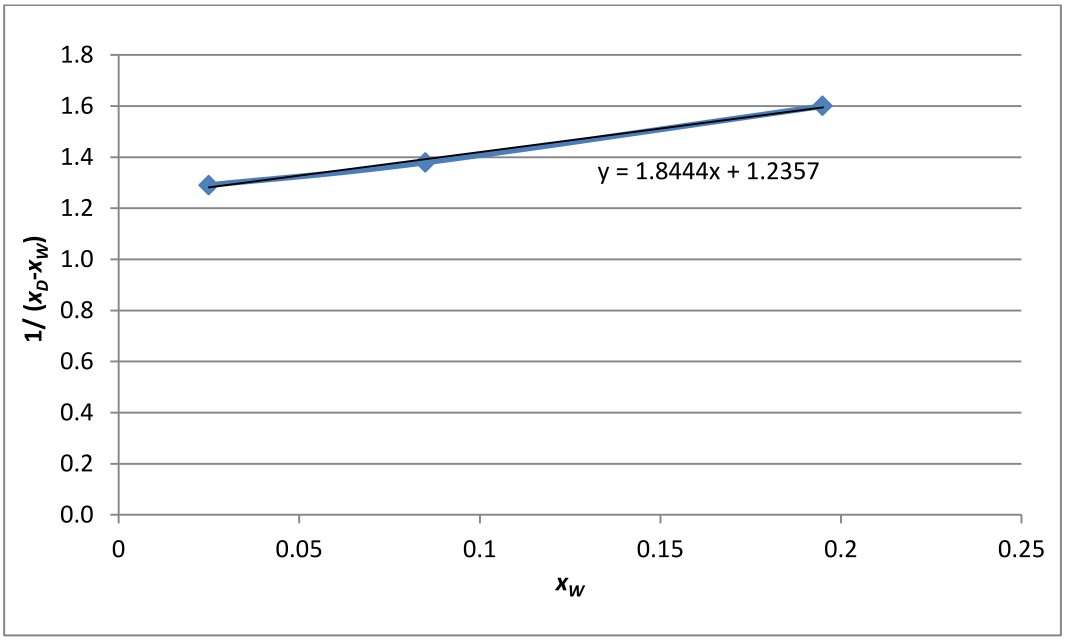


Figure 4.2: Graph of 1/ (x_D-x_W) Against x_W

$$ln\left(\frac{W_{10}}{W_0}\right) = \int_{x_{W_0}}^{x_{W_{10}}} \frac{dx_W}{x_D\text{-}x_W}$$

$$\ln\left(\frac{0.45359}{0.45913}\right) = \int_{0.0933}^{x_{W_{10}}} 1.884x_W + 1.235\ dx_W$$

$$-0.04943 = \left[\frac{1.884}{2}x^2 + 1.235x\right]_{0.09326}^{x_{W_{10}}}$$

$x_{W_{10}}$ = 0.08450.

Thus, the average ethanol composition at the distillate is

$$x_{D,avg} = \frac{W_0x_0\ -\ W_{10}x_{W_{10}}}{W_0\ -\ W_{10}} = \frac{(0.45913)(0.093257)\ -\ (0.45359)(0.08450)}{0.45913\ -\ 0.45359}$$

$x_{D,avg}$= 0.81093

Calculation at time 20 minutes, 30 minutes and 40 minutes are done based on the same method as above. The results from the calculation is tabulated in Table 4.3 and Table 4.4.

Table 4.3: Composition of Distillate and Bottoms at Constant Reflux by Graphical Method

Time, *t* (minute)	0	10	20	30	40
Ethanol mole fraction at distillate	0.0000	0.8109	0.8017	0.7998	0.7996
Water mole fraction at distillate	0.0000	0.1891	0.1983	0.2002	0.2004
Ethanol mole fraction at bottoms	0.0933	0.0845	0.0758	0.0667	0.0575
Water mole fraction at bottoms	0.9067	0.9155	0.9243	0.9333	0.9425

Table 4.4: Distillate and Bottoms Holdup at Constant Reflux by Graphical Method

Time, *t* (minutes)	0	10	20	30	40
Holdup at distillate (kmol)	0.0000	0.0055	0.0111	0.0166	0.0222
Holdup at bottoms (kmol)	0.4591	0.4536	0.4481	0.4425	0.4370

4.2.2 Simulation using Aspen Batch Distillation Software

The system parameters were defined as per Table 4.5 before running simulation in Aspen Batch Distillation. The system was set to report its simulation results every ten minutes. Table 4.6 and Table 4.7 shows the results obtained from the simulation with a constant reflux of 80 %

Table 4.5: Parameter Value/ Input for Aspen Batch Distillation

Parameter	Value/ input
Fluid Package	NRTL
Number of Stages	8
Pot orientation	Horizontal
Pot head type	Elliptical
Pot Dimension	Diameter: 0.3 m Length: 0.5 m
Condenser	Total
Reflux specification	Reflux Ratio
Heating option(Mole Boil-up Rate)	4.61354e-05 kmol/s
Total initial Charge	0.45913 kmol
Fresh charge composition(mole-fraction)	Ethanol: 0.093257 Water: 0.906743
Stages Holdups (volume)	0.001 cc

Table 4.6: Composition of Distillate and Bottoms at Constant Reflux by Aspen Batch Distillation Software

Time (minutes)	10	20	30	40
Ethanol mole fraction at distillate	0.7996	0.7990	0.7983	0.7974
Water mole fraction at distillate	0.2004	0.2010	0.2017	0.2026
Ethanol mole fraction at bottoms	0.0844	0.0752	0.0659	0.0563
Water mole fraction at bottoms	0.9156	0.9248	0.9341	0.9437

.

Table 4.7: Distillate and Bottoms Holdup at Constant Reflux by Aspen Batch Distillation Software

Time, *t* (minutes)	10	20	30	40
Holdup at distillate (kmol)	0.0057	0.0114	0.0172	0.0229
Holdup at bottoms (kmol)	0.4534	0.4477	0.4420	0.4362

4.2.3 Experimental Results by Others

Experimental results which previously done by others, was extracted to obtain the composition of ethanol at the receiver and reboiler. The average value from 15 sets of results was used as comparison with the results from ABD and graphical method. Table 4.8 shows the results for experiment with a constant reflux of 80 %. Measurement for volume was not taken due to very low amount of distillate produced in the experiments. Hence, the data for distillate and bottoms holdup is not available.

Table 4.8: Experimental Results at Constant Reflux

Time, *t* (minute)	10	20	30	40
Ethanol mole fraction at distillate	0.7400	0.7250	0.7025	0.6500
Water mole fraction at distillate	0.2600	0.2750	0.2975	0.3500
Ethanol mole fraction at bottoms	0.1063	0.0988	0.0925	0.0888
Water mole fraction at bottoms	0.8938	0.9013	0.9075	0.9113

4.2.4 Comparison between Results from Aspen Batch Distillation Simulation, Graphical Method and Experiment

The results from ABD, graphical method and experiments show a theoretical trend where ethanol purity at distillate is decreasing over time. This trend is due to unsteady state operation of batch distillation where the composition of more volatile at the bottoms is depleting over time and resulting in varying distillate composition at constant reflux (Kim & Diwekar, 2005).

Comparison of the results shows that the purity of distillate obtained by graphical methods is almost similar to results from ABD. The experimental results, however, deviated from the results from ABD and graphical methods. This deviation indicated that the experimental results are flawed, with error of 11.8 % for ethanol and 46.7 % for water composition at distillate. This large value of error is unacceptable for experimental results to be recognized even with some assumption made in simulation model. Figure 4.3, Table 4.9 and Table 4.10 shows the summary of comparison between the results from ABD, graphical method and experiments at distillate.

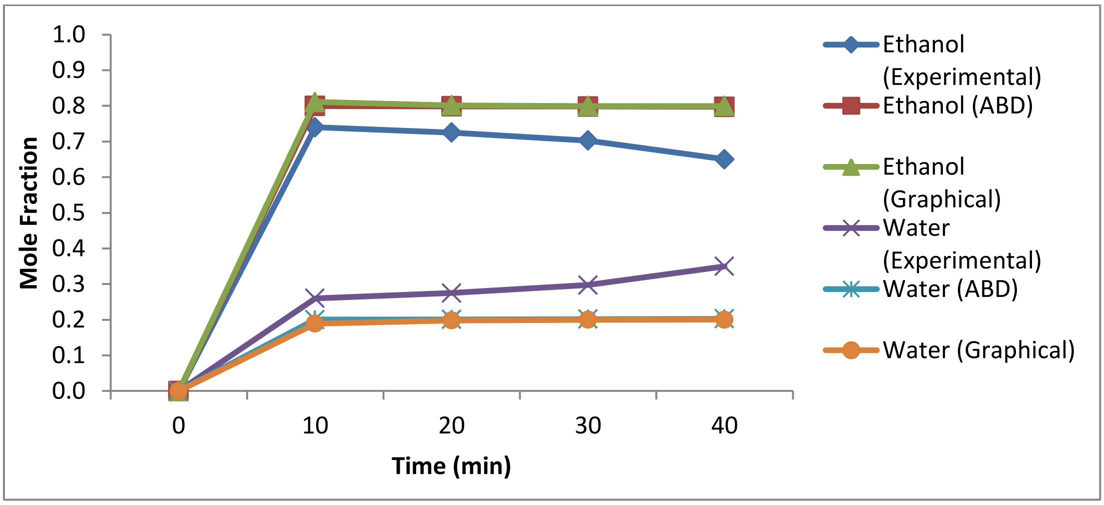


Figure 4.3: Composition of Distillate at Constant Reflux

Table 4.9: Comparison of Errors between Graphical Method and Experiment with ABD Simulation for Ethanol Distillate Composition at Constant Reflux

	Composition (mole fraction)			Percentage of error (%)	
Time (min)	Aspen Batch Distillation (basis)	Graphical Method	Experimental	Graphical Method	Experimental
10	0.7996	0.8109	0.7400	1.4171	7.4540
20	0.7990	0.8017	0.7250	0.3370	9.2579
30	0.7983	0.7998	0.7025	0.1980	11.9957
40	0.7974	0.7996	0.6500	0.2788	18.4844
			Mean	0.5577	11.7980
			SD	0.5757	4.8328

Table 4.10: Comparison of Errors between Graphical Method and Experiment with ABD Simulation for Water Distillate Composition at Constant Reflux

	Composition (mole fraction)			Percentage of error (%)	
Time (min)	Aspen Batch Distillation (basis)	Graphical Method	Experimental	Graphical Method	Experimental
10	0.2004	0.1891	0.2600	5.6542	29.7418
20	0.2010	0.1983	0.2750	1.3392	36.7941
30	0.2017	0.2002	0.2975	0.7835	47.4641
40	0.2026	0.2004	0.3500	1.0971	72.7482
			Mean	2.2185	46.6871
			SD	2.3017	18.8397

Figure 4.4 shows the bottoms composition at constant reflux. At the bottoms, the error for ethanol composition between experiment and ABD is larger (38.8 %) than between graphical method with ABD (1.1 %). Again, this large error indicates flaw in the experimental results. However, the errors for water composition are small for both experimental and graphical method, 2.8 % for experimental and 0.1 % for graphical method. One argument that can be made for this case is, even though the experimental results is presumed to be inaccurate, the effect of concentration changed of water at the bottoms is not significant because water holdup at the bottoms is much larger compared to the holdup at the distillate. Table 4.11 and Table 4.12 are the summary of percentage of errors.

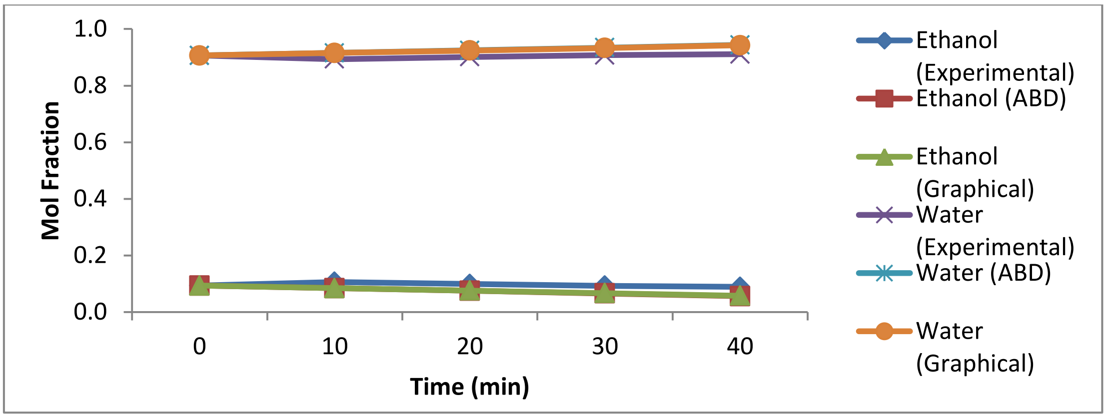


Figure 4.4: Bottoms Composition at Constant Reflux

Table 4.11: Comparison of Errors between Graphical Method and Experiment with ABD Simulation for Ethanol at Bottoms Composition at Constant Reflux

	Composition (mole fraction)			Percentage of error (%)	
Time (min)	Aspen Batch Distillation (basis)	Graphical Method	Experimental	Graphical Method	Experimental
10	0.0844	0.0845	0.1063	0.1739	25.9618
20	0.0752	0.0758	0.0988	0.6964	31.2694
30	0.0659	0.0667	0.0925	1.3065	40.4132
40	0.0563	0.0575	0.0888	2.0624	57.6377
			Mean	1.0598	38.8205
			SD	0.8130	13.8923

Table 4.12: Comparison of Errors between Graphical Method and Experiment with ABD Simulation for Water at Bottoms Composition at Constant Reflux

	Composition (mole fraction)			Percentage of error (%)	
Time (min)	Aspen Batch Distillation (basis)	Graphical Method	Experimental	Graphical Method	Experimental
10	0.9157	0.9155	0.8938	0.0160	2.3916
20	0.9248	0.9243	0.9013	0.0567	2.5437
30	0.9341	0.9333	0.9075	0.0921	2.8501
40	0.9437	0.9425	0.9113	0.1230	3.4386
			Mean	0.0720	2.8060
			SD	0.0461	0.4628

With this large error from experimental results comparing to ABD, troubleshooting was carried out on the column to identify the problems that caused the experimental results to deviate. A faulty reflux splitter of the column may explain the deviation of experimental results. The reflux splitter was found not returning the reflux back to the column at the percentage it supposes. For example, when the reflux was set at 80 % reflux, it returns only 20 % of the flow to the column, instead of 80 %. This causes the reflux to be lower than the specification. In this case, the deviation of experimental results, where the composition of ethanol in distillate is lower than those from ABD and graphical method can be explained theoretically; lower reflux ratio will produce lower purity of lighter component at the distillate for a given number of stages and reflux (Seader & Ernest, 2006; Crockett, 1986).

Comparison for holdup at distillate and bottoms is done only with both of the simulation results, since there is no experimental data. Figure 4.5 shows the results from simulation from ABD and graphical method at constant reflux of 80 %. From Figure 4.5, it can be seen that the composition obtained from graphical method is close to those from ABD with an average error of only 3.3 % at the distillate and 0.1 % at the bottoms.

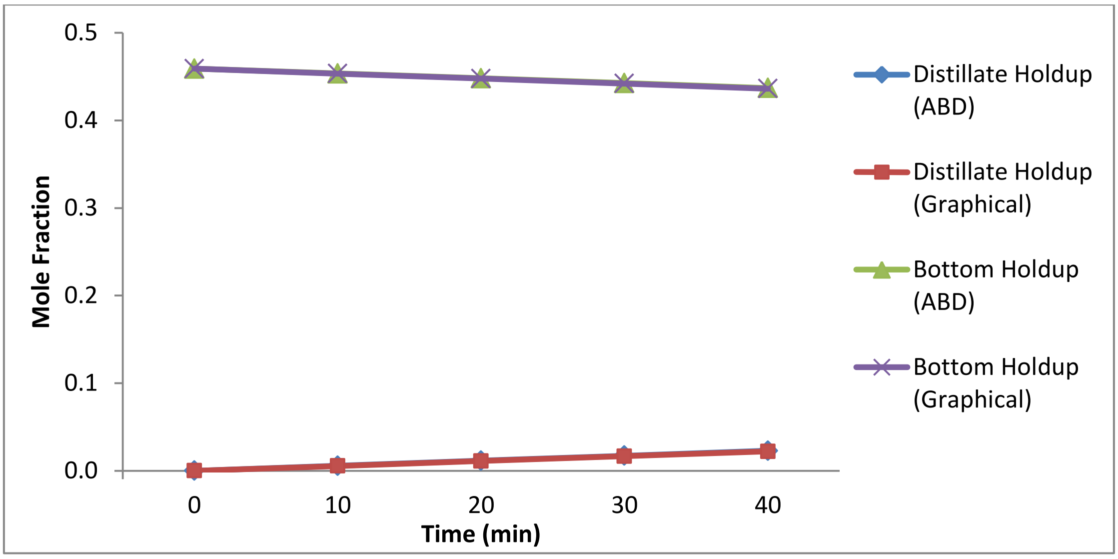


Figure 4.5: Holdup Comparison at Constant Reflux

4.3 Batch Distillation at Regulating Reflux

4.3.1 Results from Graphical Method

Simulation of regulation reflux was done by using the method described in Section 3.2.2, the time for each step is ten minutes and the reflux percentage will be increased by 10 % at the end of each step, starting with 40 % reflux. By using the boil-up rate calculated in Section 4.1 and Equation (3.12), calculation for regulating reflux is at the first step, $k = 1$, is as follow

$$W_k = W_0 - \frac{Vt}{R_k + 1}$$

$$W_1 = 0.45913 - \frac{(2.7681 \times 10^{-3})(10)}{0.66667 + 1}$$

$$W_1 = 0.4425 \text{ kmol}$$

Substituting the value of W_1 into Equation (3.8) and using the curve equation from the graph $1/(x_D - x_W)$ vs x_W for reflux 40% as shown in Table 4.13, value of x_{W_1} was calculated as follow

$$\ln\left(\frac{W_1}{W_0}\right)=\int_{x_{W_0}}^{x_{W_1}}\frac{dx_W}{x_D\text{-}x_W}$$

$$\ln\left(\frac{0.44252}{0.45913}\right)=\int_{0.0933}^{x_{W_1}}\text{-}3.253x\text{+}2.114$$

$$\text{-}0.03684=\left[\frac{\text{-}3.253}{2}\text{x}^2\text{+}2.114\text{x}\right]_{0.0933}^{x_{W_1}}$$

Table 4.13: Curve Equation at Different Reflux Percentage

Reflux Percentage (%)	Reflux Ratio	Curve Equation
40	0.66667	-3.253x + 2.114
50	1.00000	$296.4x^2$ - 47.75x + 3.460
60	1.50000	$287.9x^2$ - 38.56x + 2.656
70	2.33333	$3554.x^2$ - 236.2x + 5.21

With the aid from Microsoft Excel, the value of x_{W_1} = 0.07327 and average distillate composition for ethanol at this step can be calculated by using Equation (3.9)

$$x_{D_1}=\frac{W_0x_0\text{- }W_1x_{W_1}}{W_0\text{-}W_1}$$

$$=\frac{(0.4591)(0.0933)\text{-}(0.4425)(0.0733)}{(0.4591)\text{- }(0.4425)}$$

$$x_{D_1}\text{=}0.0626$$

and the cumulative distillate composition can be calculate by using Equation (3.10)

$$x_{D,avg}=\frac{\sum_0^1 D_1x_1}{\sum_0^1 D_1}$$

$$=\frac{(0.0626)(0.0166)}{0.0166}$$

$$x_{D,avg}=0.06257$$

Table 4.14 and Table 4.15 is the results of calculation done at reflux percentage of 40%, 50%, 60% and 70% using the modified Smoker and Rose method.

Table 4.14: Composition of Distillate and Bottoms at Regulating Reflux by Graphical Method

Reflux Percentage (%)	40	50	60	70
Ethanol mole fraction at distillate	0.6257	0.6485	0.6623	0.6698
Water mole fraction at distillate	0.3743	0.3515	0.3377	0.3302
Ethanol mole fraction at bottoms	0.0733	0.0538	0.0367	0.0189
Water mole fraction at bottoms	0.9067	0.9267	0.9462	0.9633

Table 4.15: Distillate and Bottoms Holdup at Regulating Reflux by Graphical Method

Reflux Percentage (%)	40	50	60	70
Holdup at distillate (kmol)	0.01661	0.03045	0.04152	0.05259
Holdup at bottoms (kmol)	0.44252	0.42868	0.41761	0.40654

4.3.2 Results from Aspen Batch Distillation Software

The system parameter to run the simulation is as per Table 4.5. In the simulation, four operating steps are inputted; each step will run for ten minutes, starting with a reflux ratio of 0.6667 and the reflux ratio is increased to 1.0000, 1.5000 and 2.3000 at the end of each step. These reflux ratio values indicate reflux percentage of 40 %, 50 %, 60 % and 80 % respectively. Results were obtained every ten minutes. Table 4.16 and Table 4.17 show the simulation results at regulating reflux.

Table 4.16: Composition of Distillate and Bottoms at Regulating Reflux by Aspen Batch Distillation Software

Reflux Percentage (%)	40	50	60	70
Ethanol mole fraction at distillate	0.6203	0.6325	0.6424	0.6493
Water mole fraction at distillate	0.3797	0.3675	0.3576	0.3507
Ethanol mole fraction at bottoms	0.0731	0.0540	0.0370	0.0234
Water mole fraction at bottoms	0.9269	0.9460	0.9630	0.9767

.

Table 4.17: Distillate and Bottoms Holdup at Regulating Reflux by Aspen Batch Distillation Software

Reflux Percentage (%)	40	50	60	70
Holdup at distillate (kmol)	0.0169	0.0312	0.0426	0.0513
Holdup at bottoms (kmol)	0.4422	0.4279	0.4165	0.4079

4.3.3 Experimental Results Done by Others

Five experimental results, previously done by other, were extracted to obtain the composition of ethanol at both the receiver and reboiler. Average value for reflux at 40 %, 50 %, 60 % and 70 % was tabulated in Table 4.18.

Table 4.18: Experimental Results for Regulating Reflux

Reflux Percentage (%)	40	50	60	70
Ethanol mole fraction at distillate	0.7133	0.7267	0.7567	0.8100
Water mole fraction at distillate	0.2867	0.2733	0.2433	0.1900
Ethanol mole fraction at bottoms	0.1167	0.1133	0.1067	0.1033
Water mole fraction at bottoms	0.8833	0.8867	0.8933	0.8967

4.3.4 Comparison between the Results from Aspen Batch Distillation Simulation, Graphical Method and Experiment

Figure 4.6 shows the distillate composition from ABD, graphical method and experiment at regulating reflux. From Figure 4.6, one can see that both the results from simulation and graphical method are almost the same. However, the experimental results deviate a lot from the simulation results. Error of graphical method compared with ABD for ethanol and water composition is just 2.4 % and 4.3 %, whereas, the experimental results give an error of 18.1 % for ethanol and 32.0 % for water. Table 4.19 and Table 4.20 below are the summary of comparison.

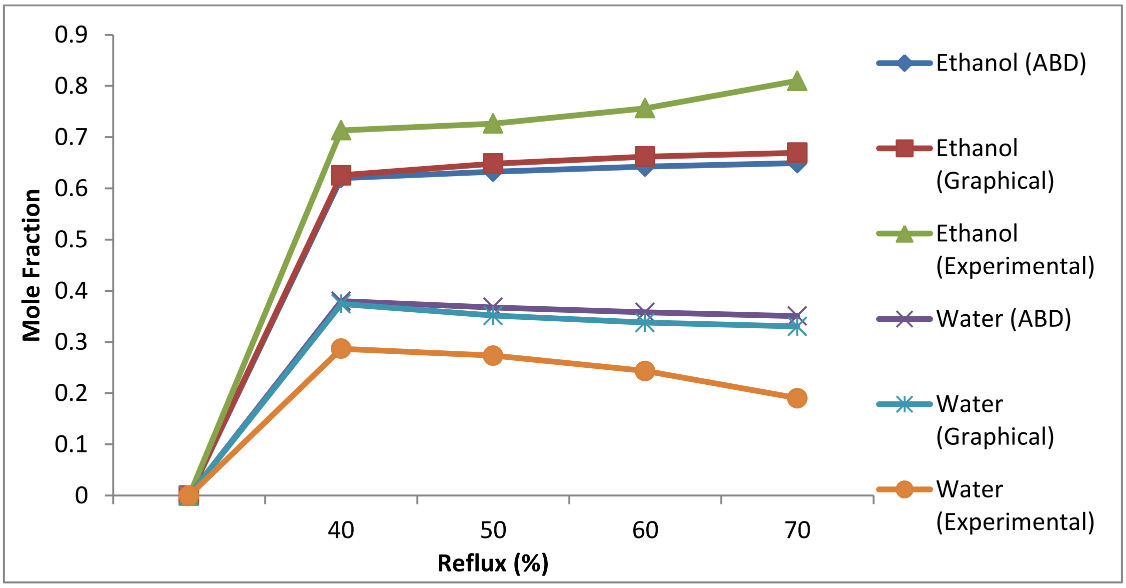


Figure 4.6: Distillate Composition at Regulating Reflux

Table 4.19: Comparison of Errors between Graphical Method and Experiment with ABD Simulation for Ethanol Distillate Composition at Regulating Reflux

	Composition (mole fraction)			Percentage of error (%)	
reflux (%)	Aspen Batch Distillation (basis)	Graphical Method	Experimental	Graphical Method	Experimental
40	0.6203	0.6257	0.7133	0.8756	15.0041
50	0.6325	0.6485	0.7267	2.5270	14.8900
60	0.6424	0.6623	0.7567	3.1015	17.7942
70	0.6493	0.6698	0.8100	3.1528	24.7451
			Mean	2.4142	18.1084
			SD	1.0642	4.6238

Table 4.20: Comparison of Errors between Graphical Method and Experiment with ABD Simulation for Water Distillate Composition at Regulating Reflux

	Composition (mole fraction)			Percentage of error (%)	
reflux (%)	Aspen Batch Distillation	Graphical Method	Experimental	Graphical Method	Experimental
40	0.3797	0.3743	0.2867	1.4303	24.5082
50	0.3675	0.3515	0.2733	4.3491	25.6258
60	0.3576	0.3377	0.2433	5.5706	31.9608
70	0.3507	0.3302	0.1900	5.8378	45.8189
			Mean	4.2969	31.9784
			SD	2.0180	9.7932

At the bottoms, errors for graphical method compared to ABD are 5.2 % for ethanol composition and 1.8 % for water composition. As can been seen from Table 4.21 there is an outlier at reflux 70 % step for graphical method with error of 19.2 %. To improve the results of graphical method, and by keeping some of the effect, winsorization is used to handle this problem (Salkind, 2010). Chincarini and Kim (2006) and Salkind (2010) explained winsorization is a process where outlier is replaced with value of the highest data point not considered as an outlier. Using winsorization, the graphical method error is impressively improved to 0.6 %. The experimental results, however, show tremendous error of 175.1 % for ethanol. Table 4.21 and Table 4.22 show comparison between the two simulation results and experimental results.

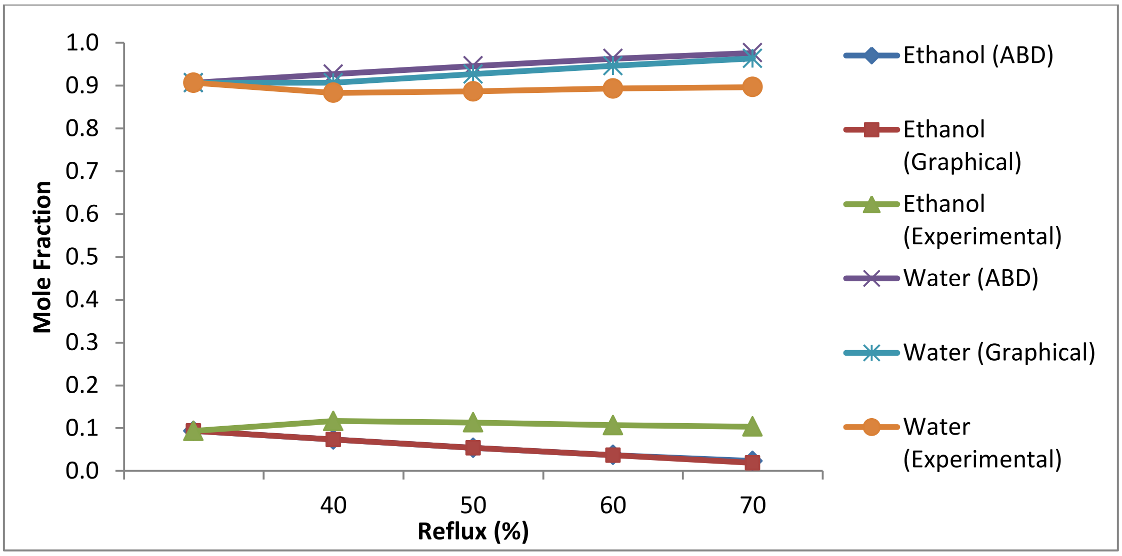


Figure 4.7: Bottoms Composition at Regulating Reflux

Table 4.21: Comparison of Errors between Graphical Method and Experiment with ABD Simulation for Ethanol at Bottoms Composition at Regulating Reflux

	Composition (mole fraction)			Percentage of error (%)	
reflux (%)	Aspen Batch Distillation	Graphical Method	Experimental	Graphical Method	Experimental
40	0.0731	0.0733	0.1167	0.2743	59.6577
50	0.0540	0.0538	0.1133	0.2725	110.0049
60	0.0370	0.0367	0.1067	0.9591	188.0158
70	0.0234	0.0189	0.1033	19.1903	342.5410
			Mean	5.1741	175.0549
			SD	9.3498	123.5147

Table 4.22: Comparison of Errors between Graphical Method and Experiment with ABD Simulation for Water at Bottoms Composition at Regulating Reflux

	Composition (mole fraction)			Percentage of error (%)	
reflux (%)	Aspen Batch Distillation	Graphical Method	Experimental	Graphical Method	Experimental
40	0.9269	0.9067	0.8833	2.1775	4.7030
50	0.9460	0.9267	0.8867	2.0408	6.2753
60	0.9630	0.9462	0.8933	1.7430	7.2310
70	0.9767	0.9633	0.8967	1.3648	8.1896
			Mean	1.8316	6.5997
			SD	0.3601	1.4865

From analysis of the data, the experimental results deviate greatly from simulation results, while the results from both graphical method and simulation are close to each other. Thus, it echoed the deduction made in Section 4.2.4, that the experimental results are erroneous.

Bottoms composition for ethanol from the experiment at constant reflux (Table 4.11) and regulating reflux (Table 4.22) shows that the composition is higher than the initial charge; opposing to the theory of batch distillation, where the composition of ethanol should decrease over time because being the lighter component, it will deplete over time (Houtman & Husain, 1955; Kim & Diwekar, 2005; Seader & Ernest, 2006; Smith & Jobson, 2000). A reason that can cause this deviation is the method used to measure the composition of the samples. The use of refractormeter to determines the composition of the sample needs a meticulous standard as the refractormeter is very sensitive to temperature, especially for most organic liquid (Williamson & Masters, 2010). Since the results are carried out by different groups of experimenters, the measurement may be made at different temperature which can affect the experiment results. This is known as "experimenter's bias". Marczyk et al. (2010) opined experimenter bias can have substantial negative impact on overall validity of a study, and to minimize this effect, dual or multiple role within the study should be minimized and standardization of all experimental procedure is required.

It is also suggested that the method for measuring the composition to be changed to a more accurate method. Brown, Foote and Brent (2011) suggested to use back titration with potassium dichromate to determine the composition, which is independent of temperature. Gas chromatography can also be employed as it is an accurate method to measure composition.

CHAPTER 5

CONCLUSION AND RECOMMENDATIONS

5.1 Conclusion

Simulation of batch distillation was successfully done using graphical method and Aspen Batch Distillation (ABD) software. At constant reflux, both the distillate and bottoms composition for ethanol drop gradually over time. The declining trend was due to the unsteady state nature of batch distillation, where the lighter component at the bottoms will deplete over time.

On the other hand, for regulating reflux, the distillate composition for ethanol was increasing as the reflux percentage increases. This is due to increasing the reflux, will result in purer product. The bottoms composition for ethanol, however, decreases over time as the lighter component is removed from the reboiler.

Comparison between the compositions obtained from graphical method with ABD shows only slight error. It has a maximum error of 5.2 %. With this minor error, the two methods supported each other and show confident in the results obtained. The experiment results, however, gives an enormous error of over 175 % compared to ABD. Responding to this large error, troubleshooting was carried out on the existing column and faulty instruments were identified. The reflux splitter was found to be faulty and was not functioning properly, causing incorrect split of reflux and distillate.

Besides that, a review on the experimental method to measure the composition used in the experiment also pointed out a weakness which will cause inaccurate results. The use of refractormeter to determine the composition requires that the temperature of all the samples is constant, which can be difficult to achieve. It is suggested to change the measuring method to either back titration with potassium dichromate or gas chromatography which is independent of temperature.

5.2 Recommendation for Future Research

In this project, simulation for regulating reflux is done by modifying Rose and Smoker (1940) method. In reality, the purpose of regulating reflux is to maintain the distillate composition throughout the batch distillation operation, as proposed by Bogart (1937). A future project on controlling the reflux ratio for binary mixture batch distillation to obtain constant distillate composition can be carried out using graphical method.

REFERENCES

AspenTech. (2008, September 8). *AspenTech Introduces aspenONE V7 for Process Engineering*. Retrieved August 24, 2011, from AspenTech: http://www.aspentech.com/uploadedFiles/News/Press_Releases/2008/AT_V7_intro_pr.pdf

Biegler, L. T., Grossmann, I. E., & Westerberg, A. W. (1997). *Systematic Methods of Chemical Process Design.* Prentice Hall.

Bogart, M. (1937). The Design Equipment For Batch Distillation Column. *American Institute of Chemical Engineers* , 139-151.

Bonsfills, A., & Puigjaner, L. (2004). Batch Distillation: Simulation and Experimental Validation. *chemical engineering and processing* , 1239-1252.

Branan, C. (2002). *Rules of Thumb for Chemical Engineers: A Manual of Quick, Accurate Solutions to Everyday Process Engineering Problems.* Gulf Professional Publishing.

Brown, W., Foote, C., & Brent, L. I. (2011). *Organic Chemistry.* Belmont, CA: Cengage Learning.

Chincarini, L. B., & Kim, D. (2006). *Quantitative Equity Portfolio Management: An Active Approach to Portfolio Construction and Management.* McGraw-Hill.

Coker, K. A. (2007). *Luwig's Applied process Design forChemical and Petrochemical Plan.* Elsevier Inc.

Cook, B. H., Engel, M. J., & Zehnder, A. J. (2004). Optimal Batch Distillation Sequences Using Aspen Plus. *Foundation of Computer-Aided Process Design 2004 : Discovery through Product and Process Design* , 259-264.

Crockett, W. (1986). *Chemical engineering: a review for the P.E. exam.* New York: John Wiley & Sons, Inc.

Distefano, G. (1968). Mathematical Modelling and Numerical Integration of Multicomponent Batch Distillation Equation. *American Institute of Chemical Engineers* , 190-199.

Diwekar, U. M., & Madhavan, K. (1991). Multicomponent Batch Distillation Design. *Industrial & Engineering Chemistry Research* , 713-721.

Diwekar, U. (1996). Understand Batch Distillation Process Principle with MultiBatchDS. *Computer Application in Engineering Education* , 275-284.

Diwekar, U., & Madhavan, K. (1991). Batch-Dist: A Comprehensive Package for Simulation, Design, Optimization and optimal Control of Multicomponent, Multifraction Batch Distillation Column. *Computers chem. Engng , 15* (12), 833-842.

Fair, J. R. (1987). *Handbook of Separation Process Technology.* (R. W. Rousseau, Ed.) John Wiley & Sons.

Greves, M. A., Mujtaba, I. M., & Hussain, M. A. (2001). Neural Networks in a Hybrid Scheme for Optimisation of Dynamic Processes: Application to Batch Distillation. In M. Hussain, & I. Mujtaba (Eds.), *Application of neural network and other learning technologies in Process Engineering* (pp. 149-171). Imperial College Press.

Houtman, J., & Husain, A. (1955). Batch Distillation. *Journal of Chemical Education* , 529-532.

Kaama, R. V., Rodríguez-Donisa, I., & Gerbaud, V. (2008). Heterogeneous Extractive Batch Distillation of Chloroform–Methanol–Water: Feasibility and Experiments. *Chemical Engineering Science* , 78-94.

Kim, K.-j., & Diwekar, U. (2005). Batch distillation. In E. Korovessi, & A. A. Linninger, *Batch Processes* (pp. 107-150). CRC Press.

Kister, H. Z. (1992). *Distillation Design.* McGraw-Hill.

Marczyk, G. R., DeMatteo, D., & Festin, D. (2010). *Essentials of Research Design and Methodology.* New Jersey: John Wiley and Sons.

Martin, J. J., & Edwards, J. B. (1964). Correlation of Latent Heat of Vaporization. *A.I.Ch.E Journal , 11* (2), 331-333.

Meadows, E. (1963). Multicomponent Batch Distillation Calculation on a Digital Computer. *Chemical Engineering Progress Symposium Series* , 48.

Mujtaba, I. (2004). *Batch Distillation Design and Operation.* London: Imperial College Press.

Rayleigh, L. (1902). On the Distillation of Binary Mixture. *Philosophical Magazine* , 521-537.

Rippin, D. W. (1983). Simulation of Single- and Multiproduct Batch Chemical Plants for Optimal Design and Operation. *Computers & Chemical Engineering* , 137-156.

Salkind, N. (2010). *Encyclopedia of Research Design* (Vol. 1). Sage Publication Inc.

Seader, J., & Ernest, J. H. (2006). *Separation Process Principles* (2nd Edition ed.). John Wiley & Sons, Inc.

Smith, R., & Jobson, M. (2000). Distillation:Overview. In I. D. Wilson, M. Cooke, C. F. Poole, & E. R. Adlard, *Encycopedia of Separation Science* (pp. 84-103). Academic Press.

Smoker, E. H., & Rose, A. (1940). Graphical Determination of Batch Distillation Curves for Binary Mixture. *American Institute of Chemical Engineers* , 285-293.

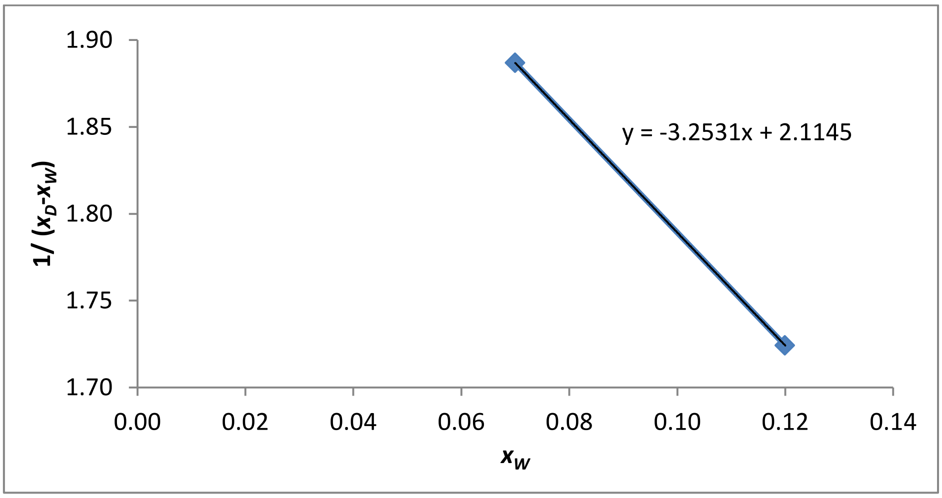
Suresh, S., & Evans, L. B. (1993). Shortcut procedure for simulating batch distillation operations. *Industrial & Engineering Chemistry Research* , 511-518.

Ugur, Y. (2002). *Multicomponent Batch Distillation Column Simulation and State Observer Design.*

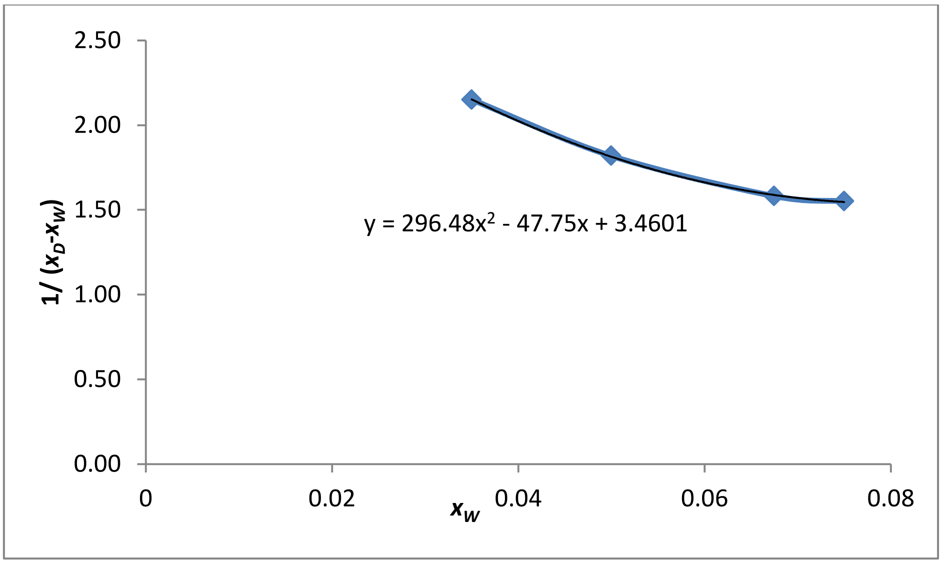
Williamson, K. L., & Masters, K. M. (2010). *Macroscale and Microscale Organic Experiments.* Belmont,CA: Cengage Learning.

APPENDICES

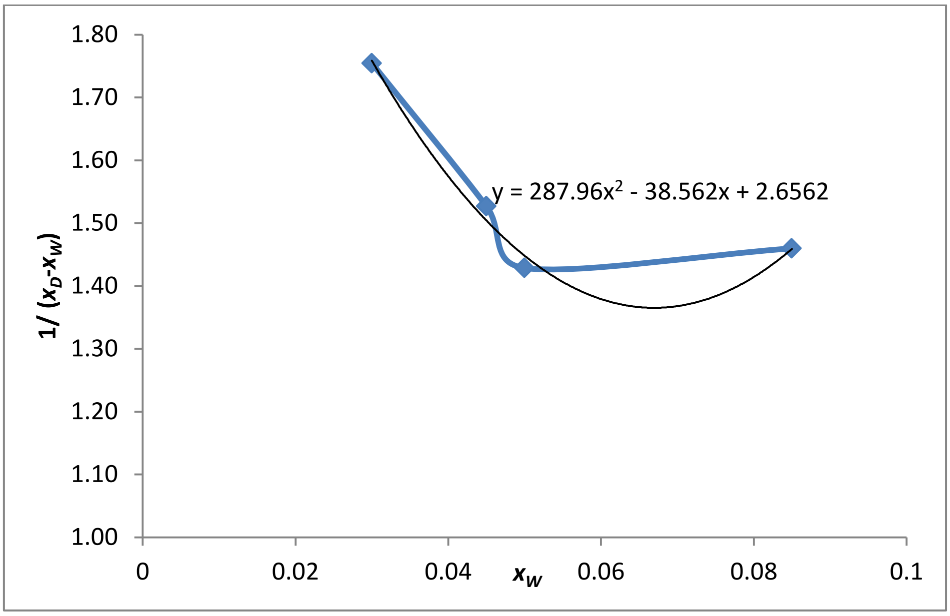
APPENDIX A: Graph of 1/ (x_D-x_W) Against x_W (regulating reflux)



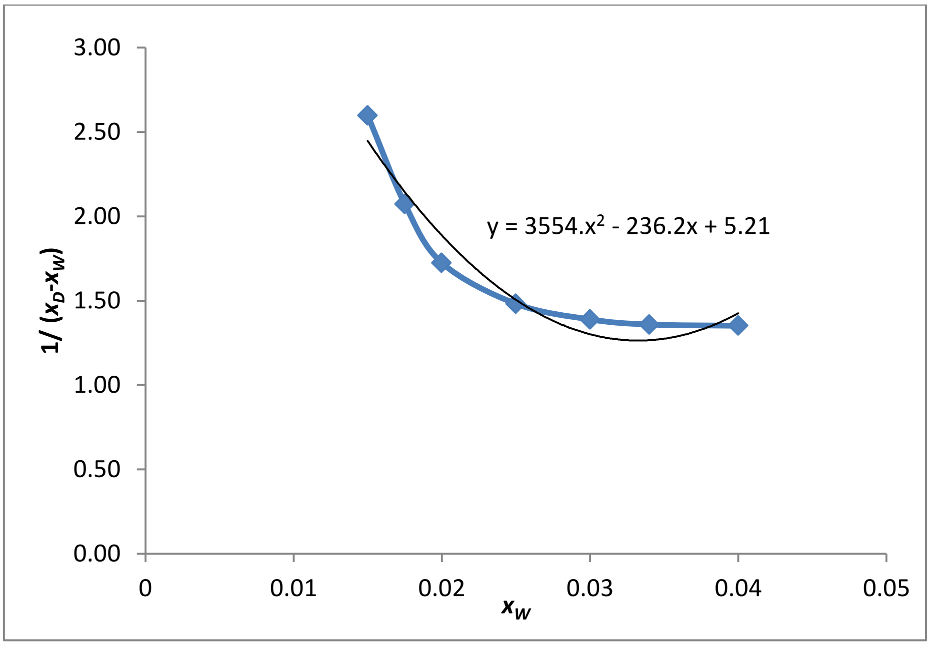
Graph of 1/ (x_D-x_W) Against x_W at reflux 40 %



Graph of 1/ (x_D-x_W) Against x_W at reflux 50 %



Graph of 1/ (x_D-x_W) Against x_W at reflux 60 %



Graph of 1/ (x_D-x_W) Against x_W at reflux 70 %

APPENDIX B: Simulation Report, Aspen Batch Distillation (Constant Reflux)

Aspen Batch Distillation Report

Report for Ronnie_Constant_Reflux.bspf
Block: B1
Simulation file: C:/Users/user/Downloads/Desktop/ronnieBatch.bspf
Report Time: Sunday, July 31, 2011 at 6:46:43 PM
Aspen Batch Distillation version: 23.0

Results

Results at: End of Operating Step : step10min
Simulation time: 0.16668 hours
Batch Number: 1

Pot Results

Holdup
Liquid level : 0.090183 m
Total molar holdup : 0.453415 kmol
Total mass holdup : 9.2414 kg
Liquid volume : 0.010475 m3
Molar boilup rate : 0.166087 kmol/hr
Mass boilup rate : 4.94183 kg/hr
Heat transfer summary
Temperature : 89.7217 C
Jacket duty : 0.012462 GJ/hr
Coils duty : -0.005585 GJ/hr
External exchanger duty : 0.0 GJ/hr
Duty for heat transfer with environment : 0.0 GJ/hr
Net duty : 0.006877 GJ/hr
Jacket
Duty : 0.012462 GJ/hr
Heat transfer area : 0.666387 m2
Heat transfer area in contact with liquid : 0.228661 m2
Medium temperature : 120 C
Coils
Heat exchanger

Profile Results

TPFQ
Basis : Mole
TPFQ Table

Stage	Temperature	Pressure	Duty	Liquid flow	Vapor flow
	C	bar	GJ/hr	kmol/hr	kmol/hr
Drum	78.2649	1.01325	0.0	0.171169	0.0
Condenser	78.2703	1.01325	-0.00675	0.136936	0.0
2	78.697	1.02754	0.0	0.136772	0.171169
3	79.1549	1.04182	0.0	0.136548	0.171006
4	79.6725	1.05611	0.0	0.136219	0.170782
5	80.3181	1.07039	0.0	0.135674	0.170453

6	81.2965	1.08468	0.0	0.134559	0.169907
7	83.4639	1.09896	0.0	0.131854	0.168793
Pot	89.7217	1.11325	0.006877	0.0	0.166087

Composition
Basis : Mole
Liquid Composition Table

Stage	ETHANOL	WATER
Drum	0.79832	0.20168
Condenser	0.79832	0.20168
2	0.771164	0.228836
3	0.738451	0.261549
4	0.695956	0.304044
5	0.634834	0.365166
6	0.532376	0.467624
7	0.319824	0.680176
Pot	0.084351	0.915649

Vapor Composition Table

Stage	ETHANOL	WATER
Drum	0.0	0.0
Condenser	0.817488	0.182512
2	0.79832	0.20168
3	0.7766	0.2234
4	0.750452	0.249548
5	0.716515	0.283485
6	0.667774	0.332226
7	0.586313	0.413687
Pot	0.418451	0.581549

Condenser Results

Main
Inlet temperature : 78.697 C
Outlet temperature : 78.2703 C
Outlet pressure : 1.01325 bar
Outlet vapor fraction : 0.0
Outlet liquid fraction : 1
Cooling
Basis : Mole
Duty : -0.00675 GJ/hr
Condensing duty : -0.00675 GJ/hr
Liquid flow rate : 0.171169 kmol/hr
Composition
Basis : Mole
Composition Table

	ETHANOL	WATER
Inlet composition	0.79832	0.20168
Liquid composition	0.79832	0.20168
Vapor composition	0.817488	0.182512

Reflux Results

Reflux
Basis : Mole
Reflux flow rate : 0.136936 kmol/hr
Reflux ratio : 4
Drum Holdup
Basis : Mole

Liquid level : 0.5 m
Liquid holdup : 1.85423e-08 kmol
Liquid volume : 1e-09 m3
Temperature : 78.2649 C
Heat transfer to environment : 0.0 GJ/hr
Drum Composition
Basis : Mole
Composition Table

	Units	ETHANOL	WATER
Component holdup	kmol	1.48027e-08	3.73962e-09
Liquid composition		0.79832	0.20168

Distillate Results

Main
Basis : Mole
Receiver taking liquid : 1
Liquid flow rate : 0.034234 kmol/hr
Composition
Basis : Mole
Composition Table

	ETHANOL	WATER
Liquid composition	0.79832	0.20168

Holdup Summary Results

Column
Column Table

	Units	Total	Pot Liquid	Tray/Packing Liquid	Reflux Drum
Total	kmol	0.453415	0.453415	1.33411e-07	1.85423e-08
Holdups					
ETHANOL	kmol	0.038248	0.038248	7.86628e-08	1.48027e-08
WATER	kmol	0.415167	0.415167	5.47487e-08	3.73962e-09
Composition					
ETHANOL	kmol/kmol	0.084351	0.084351	0.007866	0.79832
WATER	kmol/kmol	0.915649	0.915649	0.005475	0.20168

Distillate
Distillate Holdup Summary Table

	Units	Receiver 1
Inlet flow	kmol/hr	0.034234
Total holdup	kmol	0.005715
Holdup		
ETHANOL	kmol	0.004569
WATER	kmol	0.001146
Composition		
ETHANOL	kmol/kmol	0.799602
WATER	kmol/kmol	0.200398

Operating Step Results

Main
Operating Step Results Table

Step	Step End Time	Units
step10min	0.16668	hr
step20min	0.0	hr
step30min	0.0	hr
step40min	0.0	hr

Multiple Batch Results

Main

Batch number : 1
Number of batches : 1
Time from start of batch : 0.16668
Time from start of first batch : 0.16668 hr

Results

Results at: End of Operating Step : step20min
Simulation time: 0.33335 hours
Batch Number: 1

Pot Results

Holdup

Liquid level : 0.088359 m
Total molar holdup : 0.447694 kmol
Total mass holdup : 9.01021 kg
Liquid volume : 0.010171 m3
Molar boilup rate : 0.166087 kmol/hr
Mass boilup rate : 4.8542 kg/hr

Heat transfer summary

Temperature : 90.3969 C
Jacket duty : 0.012011 GJ/hr
Coils duty : -0.00511 GJ/hr
External exchanger duty : 0.0 GJ/hr
Duty for heat transfer with environment : 0.0 GJ/hr
Net duty : 0.006902 GJ/hr

Jacket

Duty : 0.012011 GJ/hr
Heat transfer area : 0.666387 m2
Heat transfer area in contact with liquid : 0.225414 m2
Medium temperature : 120 C

Coils

Heat exchanger

Profile Results

TPFQ

Basis : Mole
TPFQ Table

Stage	Temperature	Pressure	Duty	Liquid flow	Vapor flow
	C	bar	GJ/hr	kmol/hr	kmol/hr
Drum	78.2715	1.01325	0.0	0.171285	0.0
Condenser	78.274	1.01325	-0.006754	0.137028	0.0
2	78.7035	1.02754	0.0	0.136865	0.171285
3	79.1662	1.04182	0.0	0.136643	0.171122
4	79.6925	1.05611	0.0	0.136314	0.1709
5	80.3573	1.07039	0.0	0.135763	0.170571
6	81.3881	1.08468	0.0	0.13461	0.17002
7	83.7578	1.09896	0.0	0.13183	0.168867
Pot	90.3969	1.11325	0.006902	0.0	0.166087

Composition

Basis : Mole
Liquid Composition Table

Stage	ETHANOL	WATER
Drum	0.796833	0.203167

Condenser	0.796833	0.203167
2	0.769087	0.230913
3	0.73554	0.26446
4	0.691729	0.308271
5	0.628216	0.371784
6	0.520427	0.479573
7	0.296431	0.703569
Pot	0.075227	0.924773

Vapor Composition Table

Stage	ETHANOL	WATER
Drum	0.0	0.0
Condenser	0.816367	0.183633
2	0.796833	0.203167
3	0.774642	0.225358
4	0.747826	0.252174
5	0.712838	0.287162
6	0.66219	0.33781
7	0.576499	0.423501
Pot	0.399642	0.600358

Condenser Results

Main
Inlet temperature : 78.7035 C
Outlet temperature : 78.274 C
Outlet pressure : 1.01325 bar
Outlet vapor fraction : 0.0
Outlet liquid fraction : 1
Cooling
Basis : Mole
Duty : -0.006754 GJ/hr
Condensing duty : -0.006754 GJ/hr
Liquid flow rate : 0.171285 kmol/hr
Composition
Basis : Mole
Composition Table

	ETHANOL	WATER
Inlet composition	0.796833	0.203167
Liquid composition	0.796833	0.203167
Vapor composition	0.816367	0.183633

Reflux Results

Reflux
Basis : Mole
Reflux flow rate : 0.137028 kmol/hr
Reflux ratio : 4
Drum Holdup
Basis : Mole
Liquid level : 0.5 m
Liquid holdup : 1.8564e-08 kmol
Liquid volume : 1e-09 m3
Temperature : 78.2715 C
Heat transfer to environment : 0.0 GJ/hr
Drum Composition
Basis : Mole
Composition Table

	Units	ETHANOL	WATER
Component holdup	kmol	1.47924e-08	3.7716e-09

Liquid composition		0.796833	0.203167

Distillate Results

Main
Basis : Mole
Receiver taking liquid : 1
Liquid flow rate : 0.034257 kmol/hr
Composition
Basis : Mole
Composition Table

	ETHANOL	WATER
Liquid composition	0.796833	0.203167

Holdup Summary Results

Column
Column Table

	Units	Total	Pot Liquid	Tray/Packing Liquid	Reflux Drum
Total	kmol	0.447694	0.447694	1.34911e-07	1.8564e-08
Holdups					
ETHANOL	kmol	0.033681	0.033681	7.7973e-08	1.47924e-08
WATER	kmol	0.414014	0.414013	5.69377e-08	3.7716e-09
Composition					
ETHANOL	kmol/kmol	0.075227	0.075227	0.007797	0.796833
WATER	kmol/kmol	0.924773	0.924773	0.005694	0.203167

Distillate
Distillate Holdup Summary Table

	Units	Receiver 1
Inlet flow	kmol/hr	0.034257
Total holdup	kmol	0.011436
Holdup		
ETHANOL	kmol	0.009137
WATER	kmol	0.002299
Composition		
ETHANOL	kmol/kmol	0.798968
WATER	kmol/kmol	0.201032

Operating Step Results

Main
Operating Step Results Table

Step	Step End Time	Units
step10min	0.16668	hr
step20min	0.33335	hr
step30min	0.0	hr
step40min	0.0	hr

Multiple Batch Results

Main
Batch number : 1
Number of batches : 1
Time from start of batch : 0.33335
Time from start of first batch : 0.33335 hr

Results

Results at: End of Operating Step : step30min
Simulation time: 0.50002 hours
Batch Number: 1

Pot Results

Holdup
Liquid level : 0.086528 m
Total molar holdup : 0.441968 kmol
Total mass holdup : 8.77903 kg
Liquid volume : 0.009868 m3
Molar boilup rate : 0.166087 kmol/hr
Mass boilup rate : 4.75025 kg/hr
Heat transfer summary
Temperature : 91.1876 C
Jacket duty : 0.011521 GJ/hr
Coils duty : -0.004593 GJ/hr
External exchanger duty : 0.0 GJ/hr
Duty for heat transfer with environment : 0.0 GJ/hr
Net duty : 0.006928 GJ/hr
Jacket
Duty : 0.011521 GJ/hr
Heat transfer area : 0.666387 m2
Heat transfer area in contact with liquid : 0.222145 m2
Medium temperature : 120 C
Coils
Heat exchanger

Profile Results

TPFQ
Basis : Mole
TPFQ Table

Stage	Temperature	Pressure	Duty	Liquid flow	Vapor flow
	C	bar	GJ/hr	kmol/hr	kmol/hr
Drum	78.276	1.01325	0.0	0.171376	0.0
Condenser	78.2789	1.01325	-0.006757	0.1371	0.0
2	78.712	1.02754	0.0	0.136942	0.171376
3	79.1809	1.04182	0.0	0.136725	0.171217
4	79.7188	1.05611	0.0	0.1364	0.171
5	80.4088	1.07039	0.0	0.135848	0.170675
6	81.5103	1.08468	0.0	0.134656	0.170123
7	84.1504	1.09896	0.0	0.131812	0.168932
Pot	91.1876	1.11325	0.006928	0.0	0.166087

Composition
Basis : Mole
Liquid Composition Table

Stage	ETHANOL	WATER
Drum	0.794925	0.205075
Condenser	0.794925	0.205075
2	0.766433	0.233567
3	0.731832	0.268168
4	0.68636	0.31364
5	0.619819	0.380181
6	0.505262	0.494738
7	0.268748	0.731252
Pot	0.065877	0.934123

Vapor Composition Table

Stage	ETHANOL	WATER
Drum	0.0	0.0
Condenser	0.814921	0.185079
2	0.794925	0.205075
3	0.772137	0.227863
4	0.744478	0.255522
5	0.708162	0.291838
6	0.655098	0.344902
7	0.564032	0.435968
Pot	0.377333	0.622667

Condenser Results

Main
Inlet temperature : 78.712 C
Outlet temperature : 78.2789 C
Outlet pressure : 1.01325 bar
Outlet vapor fraction : 0.0
Outlet liquid fraction : 1
Cooling
Basis : Mole
Duty : -0.006757 GJ/hr
Condensing duty : -0.006757 GJ/hr
Liquid flow rate : 0.171376 kmol/hr
Composition
Basis : Mole
Composition Table

	ETHANOL	WATER
Inlet composition	0.794925	0.205075
Liquid composition	0.794925	0.205075
Vapor composition	0.814921	0.185079

Reflux Results

Reflux
Basis : Mole
Reflux flow rate : 0.1371 kmol/hr
Reflux ratio : 4
Drum Holdup
Basis : Mole
Liquid level : 0.5 m
Liquid holdup : 1.85919e-08 kmol
Liquid volume : 1e-09 m3
Temperature : 78.276 C
Heat transfer to environment : 0.0 GJ/hr
Drum Composition
Basis : Mole
Composition Table

	Units	ETHANOL	WATER
Component holdup	kmol	1.47792e-08	3.81276e-09
Liquid composition		0.794925	0.205075

Distillate Results

Main
Basis : Mole
Receiver taking liquid : 1
Liquid flow rate : 0.034275 kmol/hr

Composition
Basis : Mole
Composition Table

	ETHANOL	WATER
Liquid composition	0.794925	0.205075

Holdup Summary Results

Column
Column Table

	Units	Total	Pot Liquid	Tray/Packing Liquid	Reflux Drum
Total	kmol	0.441968	0.441968	1.3682e-07	1.85919e-08
Holdups					
ETHANOL	kmol	0.029118	0.029118	7.70895e-08	1.47792e-08
WATER	kmol	0.41285	0.41285	5.97306e-08	3.81276e-09
Composition					
ETHANOL	kmol/kmol	0.065878	0.065877	0.007709	0.794925
WATER	kmol/kmol	0.934122	0.934123	0.005973	0.205075

Distillate
Distillate Holdup Summary Table

	Units	Receiver 1
Inlet flow	kmol/hr	0.034275
Total holdup	kmol	0.017163
Holdup		
ETHANOL	kmol	0.0137
WATER	kmol	0.003463
Composition		
ETHANOL	kmol/kmol	0.798256
WATER	kmol/kmol	0.201744

Operating Step Results

Main
Operating Step Results Table

Step	Step End Time	Units
step10min	0.16668	hr
step20min	0.33335	hr
step30min	0.50002	hr
step40min	0.0	hr

Multiple Batch Results

Main
Batch number : 1
Number of batches : 1
Time from start of batch : 0.50002
Time from start of first batch : 0.50002 hr

Results

Results at: End of Operating Step : step40min
Simulation time: 0.666689 hours
Batch Number: 1

Pot Results

Holdup
Liquid level : 0.084691 m

Total molar holdup : 0.436236 kmol
Total mass holdup : 8.54797 kg
Liquid volume : 0.009566 m3
Molar boilup rate : 0.166087 kmol/hr
Mass boilup rate : 4.62513 kg/hr
Heat transfer summary
Temperature : 92.1229 C
Jacket duty : 0.010982 GJ/hr
Coils duty : -0.004024 GJ/hr
External exchanger duty : 0.0 GJ/hr
Duty for heat transfer with environment : 0.0 GJ/hr
Net duty : 0.006958 GJ/hr
Jacket
Duty : 0.010982 GJ/hr
Heat transfer area : 0.666387 m2
Heat transfer area in contact with liquid : 0.218857 m2
Medium temperature : 120 C
Coils
Heat exchanger

Profile Results

TPFQ
Basis : Mole
TPFQ Table

Stage	Temperature	Pressure	Duty	Liquid flow	Vapor flow
	C	bar	GJ/hr	kmol/hr	kmol/hr
Drum	78.2819	1.01325	0.0	0.171414	0.0
Condenser	78.2858	1.01325	-0.006756	0.137131	0.0
2	78.724	1.02754	0.0	0.136983	0.171414
3	79.2015	1.04182	0.0	0.136776	0.171265
4	79.7559	1.05611	0.0	0.136464	0.171059
5	80.482	1.07039	0.0	0.13592	0.170747
6	81.6859	1.08468	0.0	0.134699	0.170203
7	84.7064	1.09896	0.0	0.131805	0.168982
Pot	92.1229	1.11325	0.006958	0.0	0.166087

Composition
Basis : Mole
Liquid Composition Table

Stage	ETHANOL	WATER
Drum	0.792312	0.207688
Condenser	0.792312	0.207688
2	0.762817	0.237183
3	0.726801	0.273199
4	0.6791	0.3209
5	0.608498	0.391502
6	0.484895	0.515105
7	0.23556	0.76444
Pot	0.0563	0.9437

Vapor Composition Table

Stage	ETHANOL	WATER
Drum	0.0	0.0
Condenser	0.812932	0.187068
2	0.792312	0.207688
3	0.768722	0.231278
4	0.73993	0.26007
5	0.701831	0.298169
6	0.645522	0.354478

7	0.547262	0.452738
Pot	0.350479	0.649521

Condenser Results

Main
Inlet temperature : 78.724 C
Outlet temperature : 78.2858 C
Outlet pressure : 1.01325 bar
Outlet vapor fraction : 0.0
Outlet liquid fraction : 1
Cooling
Basis : Mole
Duty : -0.006756 GJ/hr
Condensing duty : -0.006756 GJ/hr
Liquid flow rate : 0.171414 kmol/hr
Composition
Basis : Mole
Composition Table

	ETHANOL	WATER
Inlet composition	0.792312	0.207688
Liquid composition	0.792312	0.207688
Vapor composition	0.812932	0.187068

Reflux Results

Reflux
Basis : Mole
Reflux flow rate : 0.137131 kmol/hr
Reflux ratio : 4
Drum Holdup
Basis : Mole
Liquid level : 0.5 m
Liquid holdup : 1.86302e-08 kmol
Liquid volume : 1e-09 m3
Temperature : 78.2819 C
Heat transfer to environment : 0.0 GJ/hr
Drum Composition
Basis : Mole
Composition Table

	Units	ETHANOL	WATER
Component holdup	kmol	1.47609e-08	3.86929e-09
Liquid composition		0.792312	0.207688

Distillate Results

Main
Basis : Mole
Receiver taking liquid : 1
Liquid flow rate : 0.034283 kmol/hr
Composition
Basis : Mole
Composition Table

	ETHANOL	WATER
Liquid composition	0.792312	0.207688

Holdup Summary Results

Column
Column Table

	Units	Total	Pot Liquid	Tray/Packing Liquid	Reflux Drum
Total	kmol	0.436236	0.436236	1.39358e-07	1.86302e-08
Holdups					
ETHANOL	kmol	0.024562	0.024562	7.59067e-08	1.47609e-08
WATER	kmol	0.411674	0.411674	6.34513e-08	3.86929e-09
Composition					
ETHANOL	kmol/kmol	0.0563	0.0563	0.007591	0.792312
WATER	kmol/kmol	0.9437	0.9437	0.006345	0.207688

Distillate

Distillate Holdup Summary Table

	Units	Receiver 1
Inlet flow	kmol/hr	0.034283
Total holdup	kmol	0.022895
Holdup		
ETHANOL	kmol	0.018256
WATER	kmol	0.004639
Composition		
ETHANOL	kmol/kmol	0.797393
WATER	kmol/kmol	0.202607

Operating Step Results

Main

Operating Step Results Table

Step	Step End Time	Units
step10min	0.16668	hr
step20min	0.33335	hr
step30min	0.50002	hr
step40min	0.666689	hr

Multiple Batch Results

Main

Batch number : 1

Number of batches : 1

Time from start of batch : 0.666689

Time from start of first batch : 0.666689 hr

Results

Results at: End of Batch

Simulation time: 0.666689 hours

Batch Number: 1

Results reported for last step

APPENDIX C: Result, Aspen Batch Distillation (Regulating Reflux)

Aspen Batch Distillation Report

Report for Ronnie_Regulating_Reflux.bspf
Block: B1
Simulation file: C:/Users/user/Downloads/Desktop/ronnieRE.bspf
Report Time: Monday, August 01, 2011 at 4:12:11 PM
Aspen Batch Distillation version: 23.0

Results

Results at: End of Operating Step : Reflux_40
Simulation time: 0.16668 hours
Batch Number: 1

Pot Results

Holdup
Liquid level : 0.087364 m
Total molar holdup : 0.442184 kmol
Total mass holdup : 8.87269 kg
Liquid volume : 0.010006 m3
Molar boilup rate : 0.166087 kmol/hr
Mass boilup rate : 4.83134 kg/hr
Heat transfer summary
Temperature : 90.5716 C
Jacket duty : 0.011847 GJ/hr
Coils duty : -0.004777 GJ/hr
External exchanger duty : 0.0 GJ/hr
Duty for heat transfer with environment : 0.0 GJ/hr
Net duty : 0.007069 GJ/hr
Jacket
Duty : 0.011847 GJ/hr
Heat transfer area : 0.666387 m2
Heat transfer area in contact with liquid : 0.223638 m2
Medium temperature : 120 C
Coils
Heat exchanger

Profile Results

TPFQ
Basis : Mole
TPFQ Table

Stage	Temperature	Pressure	Duty	Liquid flow	Vapor flow
	C	bar	GJ/hr	kmol/hr	kmol/hr
Drum	79.0544	1.01325	0.0	0.169615	0.0
Condenser	79.2059	1.01325	-0.006704	0.067846	0.0
2	81.4063	1.02754	0.0	0.067316	0.169615
3	84.5213	1.04182	0.0	0.066222	0.169086
4	87.5955	1.05611	0.0	0.065013	0.167991
5	89.1689	1.07039	0.0	0.064523	0.166782

6	89.8157	1.08468	0.0	0.064372	0.166292
7	90.2184	1.09896	0.0	0.064318	0.166142
Pot	90.5716	1.11325	0.007069	0.0	0.166087

Composition

Basis : Mole

Liquid Composition Table

Stage	ETHANOL	WATER
Drum	0.596197	0.403803
Condenser	0.596197	0.403803
2	0.38484	0.61516
3	0.196622	0.803378
4	0.105614	0.894386
5	0.081844	0.918156
6	0.076916	0.923084
7	0.075971	0.924029
Pot	0.073073	0.926927

Vapor Composition Table

Stage	ETHANOL	WATER
Drum	0.0	0.0
Condenser	0.692237	0.307763
2	0.596197	0.403803
3	0.51205	0.48795
4	0.438685	0.561315
5	0.404965	0.595035
6	0.396624	0.603376
7	0.394998	0.605002
Pot	0.394737	0.605263

Condenser Results

Main

Inlet temperature : 81.4063 C

Outlet temperature : 79.2059 C

Outlet pressure : 1.01325 bar

Outlet vapor fraction : 0.0

Outlet liquid fraction : 1

Cooling

Basis : Mole

Duty : -0.006704 GJ/hr

Condensing duty : -0.006704 GJ/hr

Liquid flow rate : 0.169615 kmol/hr

Composition

Basis : Mole

Composition Table

	ETHANOL	WATER
Inlet composition	0.596197	0.403803
Liquid composition	0.596197	0.403803
Vapor composition	0.692237	0.307763

Reflux Results

Reflux

Basis : Mole

Reflux flow rate : 0.067846 kmol/hr

Reflux ratio : 0.66666

Drum Holdup

Basis : Mole

Liquid level : 0.5 m

Liquid holdup : 2.20992e-08 kmol
Liquid volume : 1e-09 m3
Temperature : 79.0544 C
Heat transfer to environment : 0.0 GJ/hr
<u>Drum Composition</u>
Basis : Mole
Composition Table

	Units	ETHANOL	WATER
Component holdup	kmol	1.31742e-08	8.92504e-09
Liquid composition		0.596197	0.403803

Distillate Results

<u>Main</u>
Basis : Mole
Receiver taking liquid : 1
Liquid flow rate : 0.10177 kmol/hr
<u>Composition</u>
Basis : Mole
Composition Table

	ETHANOL	WATER
Liquid composition	0.596197	0.403803

Holdup Summary Results

<u>Column</u>
Column Table

	Units	Total	Pot Liquid	Tray/Packing Liquid	Reflux Drum
Total	kmol	0.442184	0.442184	2.35548e-07	2.20992e-08
Holdups					
ETHANOL	kmol	0.032318	0.032318	3.22112e-08	1.31742e-08
WATER	kmol	0.409866	0.409866	2.03337e-07	8.92504e-09
Composition					
ETHANOL	kmol/kmol	0.073073	0.073073	0.003221	0.596197
WATER	kmol/kmol	0.926927	0.926927	0.020334	0.403803

<u>Distillate</u>
Distillate Holdup Summary Table

	Units	Receiver 1
Inlet flow	kmol/hr	0.10177
Total holdup	kmol	0.016946
Holdup		
ETHANOL	kmol	0.010501
WATER	kmol	0.006445
Composition		
ETHANOL	kmol/kmol	0.620268
WATER	kmol/kmol	0.379732

Operating Step Results

<u>Main</u>
Operating Step Results Table

Step	Step End Time	Units
Reflux_40	0.16668	hr
Reflux_50	0.0	hr
Reflux_60	0.0	hr
Reflux_70	0.0	hr
Reflux_80	0.0	hr

Multiple Batch Results
Main
Batch number : 1
Number of batches : 1
Time from start of batch : 0.16668
Time from start of first batch : 0.16668 hr

Results

Results at: End of Operating Step : Reflux_50
Simulation time: 0.33335 hours
Batch Number: 1

Pot Results
Holdup
Liquid level : 0.083324 m
Total molar holdup : 0.427948 kmol
Total mass holdup : 8.35767 kg
Liquid volume : 0.009343 m3
Molar boilup rate : 0.166087 kmol/hr
Mass boilup rate : 4.59069 kg/hr
Heat transfer summary
Temperature : 92.3766 C
Jacket duty : 0.010761 GJ/hr
Coils duty : -0.003625 GJ/hr
External exchanger duty : 0.0 GJ/hr
Duty for heat transfer with environment : 0.0 GJ/hr
Net duty : 0.007136 GJ/hr
Jacket
Duty : 0.010761 GJ/hr
Heat transfer area : 0.666387 m2
Heat transfer area in contact with liquid : 0.216405 m2
Medium temperature : 120 C
Coils
Heat exchanger

Profile Results
TPFQ
Basis : Mole
TPFQ Table

Stage	Temperature	Pressure	Duty	Liquid flow	Vapor flow
	C	bar	GJ/hr	kmol/hr	kmol/hr
Drum	79.0156	1.01325	0.0	0.17117	0.0
Condenser	79.1422	1.01325	-0.006676	0.085585	0.0
2	80.8808	1.02754	0.0	0.086096	0.17117
3	83.4744	1.04182	0.0	0.086196	0.171681
4	87.0225	1.05611	0.0	0.084235	0.171782
5	90.0622	1.07039	0.0	0.081663	0.16982
6	91.4305	1.08468	0.0	0.080732	0.167248
7	91.9922	1.09896	0.0	0.080502	0.166317
Pot	92.3766	1.11325	0.007136	0.0	0.166087

Composition
Basis : Mole
Liquid Composition Table

Stage	ETHANOL	WATER
Drum	0.612552	0.387448

Condenser	0.612552	0.387448
2	0.454101	0.545899
3	0.288182	0.711818
4	0.141503	0.858497
5	0.07536	0.92464
6	0.059583	0.940417
7	0.056626	0.943374
Pot	0.053967	0.946033

Vapor Composition Table

Stage	ETHANOL	WATER
Drum	0.0	0.0
Condenser	0.695354	0.304646
2	0.612552	0.387448
3	0.533084	0.466916
4	0.449775	0.550225
5	0.378898	0.621102
6	0.350251	0.649749
7	0.344129	0.655871
Pot	0.343088	0.656912

Condenser Results

Main
Inlet temperature : 80.8808 C
Outlet temperature : 79.1422 C
Outlet pressure : 1.01325 bar
Outlet vapor fraction : 0.0
Outlet liquid fraction : 1
Cooling
Basis : Mole
Duty : -0.006676 GJ/hr
Condensing duty : -0.006676 GJ/hr
Liquid flow rate : 0.17117 kmol/hr
Composition
Basis : Mole
Composition Table

	ETHANOL	WATER
Inlet composition	0.612552	0.387448
Liquid composition	0.612552	0.387448
Vapor composition	0.695354	0.304646

Reflux Results

Reflux
Basis : Mole
Reflux flow rate : 0.085585 kmol/hr
Reflux ratio : 1
Drum Holdup
Basis : Mole
Liquid level : 0.5 m
Liquid holdup : 2.17149e-08 kmol
Liquid volume : 1e-09 m3
Temperature : 79.0156 C
Heat transfer to environment : 0.0 GJ/hr
Drum Composition
Basis : Mole
Composition Table

	Units	ETHANOL	WATER

Component holdup	kmol	1.32974e-08	8.41746e-09
Liquid composition		0.612552	0.387448

Distillate Results

Main
Basis : Mole
Receiver taking liquid : 1
Liquid flow rate : 0.085585 kmol/hr
Composition
Basis : Mole
Composition Table

	ETHANOL	WATER
Liquid composition	0.612552	0.387448

Holdup Summary Results

Column
Column Table

	Units	Total	Pot Liquid	Tray/Packing Liquid	Reflux Drum
Total	kmol	0.427948	0.427948	2.28961e-07	2.17149e-08
Holdups					
ETHANOL	kmol	0.023101	0.023101	3.43577e-08	1.32974e-08
WATER	kmol	0.404847	0.404847	1.94603e-07	8.41746e-09
Composition					
ETHANOL	kmol/kmol	0.053967	0.053967	0.003436	0.612552
WATER	kmol/kmol	0.946033	0.946033	0.01946	0.387448

Distillate
Distillate Holdup Summary Table

	Units	Receiver 1
Inlet flow	kmol/hr	0.085585
Total holdup	kmol	0.031182
Holdup		
ETHANOL	kmol	0.019718
WATER	kmol	0.011464
Composition		
ETHANOL	kmol/kmol	0.632489
WATER	kmol/kmol	0.367511

Operating Step Results

Main
Operating Step Results Table

Step	Step End Time	Units
Reflux_40	0.16668	hr
Reflux_50	0.33335	hr
Reflux_60	0.0	hr
Reflux_70	0.0	hr
Reflux_80	0.0	hr

Multiple Batch Results

Main
Batch number : 1
Number of batches : 1
Time from start of batch : 0.33335
Time from start of first batch : 0.33335 hr

Results

Results at: End of Operating Step : Reflux_60
Simulation time: 0.500017 hours
Batch Number: 1

Pot Results

Holdup
Liquid level : 0.07998 m
Total molar holdup : 0.416484 kmol
Total mass holdup : 7.93578 kg
Liquid volume : 0.008803 m3
Molar boilup rate : 0.166087 kmol/hr
Mass boilup rate : 4.29354 kg/hr

Heat transfer summary
Temperature : 94.5111 C
Jacket duty : 0.009652 GJ/hr
Coils duty : -0.002493 GJ/hr
External exchanger duty : 0.0 GJ/hr
Duty for heat transfer with environment : 0.0 GJ/hr
Net duty : 0.007159 GJ/hr

Jacket
Duty : 0.009652 GJ/hr
Heat transfer area : 0.666387 m2
Heat transfer area in contact with liquid : 0.210383 m2
Medium temperature : 120 C

Coils

Heat exchanger

Profile Results

TPFQ
Basis : Mole
TPFQ Table

Stage	Temperature	Pressure	Duty	Liquid flow	Vapor flow
	C	bar	GJ/hr	kmol/hr	kmol/hr
Drum	78.9543	1.01325	0.0	0.169933	0.0
Condenser	78.9425	1.01325	-0.006805	0.10196	0.0
2	80.6671	1.02754	0.0	0.100283	0.169933
3	84.6177	1.04182	0.0	0.099498	0.168256
4	90.346	1.05611	0.0	0.09849	0.167474
5	92.9836	1.07039	0.0	0.098155	0.166467
6	93.7432	1.08468	0.0	0.09811	0.166132
7	94.1535	1.09896	0.0	0.09811	0.166087
Pot	94.5111	1.11325	0.007159	0.0	0.166087

Composition
Basis : Mole
Liquid Composition Table

Stage	ETHANOL	WATER
Drum	0.628947	0.371053
Condenser	0.628947	0.371053
2	0.430812	0.569188
3	0.17864	0.82136
4	0.062287	0.937713
5	0.040601	0.959399
6	0.037524	0.962476
7	0.037105	0.962895
Pot	0.037035	0.962965

Vapor Composition Table

Stage	ETHANOL	WATER
Drum	0.0	0.0
Condenser	0.715517	0.284483
2	0.628947	0.371053
3	0.510854	0.489146
4	0.36139	0.63861
5	0.29366	0.70634
6	0.281315	0.718685
7	0.279562	0.720438
Pot	0.279314	0.720686

Condenser Results

Main
Inlet temperature : 80.6671 C
Outlet temperature : 78.9425 C
Outlet pressure : 1.01325 bar
Outlet vapor fraction : 0.0
Outlet liquid fraction : 1
Cooling
Basis : Mole
Duty : -0.006805 GJ/hr
Condensing duty : -0.006805 GJ/hr
Liquid flow rate : 0.169933 kmol/hr
Composition
Basis : Mole
Composition Table

	ETHANOL	WATER
Inlet composition	0.628947	0.371053
Liquid composition	0.628947	0.371053
Vapor composition	0.715517	0.284483

Reflux Results

Reflux
Basis : Mole
Reflux flow rate : 0.10196 kmol/hr
Reflux ratio : 1.5
Drum Holdup
Basis : Mole
Liquid level : 0.5 m
Liquid holdup : 2.14832e-08 kmol
Liquid volume : 1e-09 m3
Temperature : 78.9543 C
Heat transfer to environment : 0.0 GJ/hr
Drum Composition
Basis : Mole
Composition Table

	Units	ETHANOL	WATER
Component holdup	kmol	1.35118e-08	7.97139e-09
Liquid composition		0.628947	0.371053

Distillate Results

Main
Basis : Mole

Receiver taking liquid : 1
Liquid flow rate : 0.067973 kmol/hr
Composition
Basis : Mole
Composition Table

	ETHANOL	WATER
Liquid composition	0.628947	0.371053

Holdup Summary Results

Column
Column Table

	Units	Total	Pot Liquid	Tray/Packing Liquid	Reflux Drum
Total	kmol	0.416484	0.416484	2.49656e-07	2.14832e-08
Holdups					
ETHANOL	kmol	0.015424	0.015424	2.61326e-08	1.35118e-08
WATER	kmol	0.40106	0.401059	2.23524e-07	7.97139e-09
Composition					
ETHANOL	kmol/kmol	0.037035	0.037035	0.002613	0.628947
WATER	kmol/kmol	0.962965	0.962965	0.022352	0.371053

Distillate
Distillate Holdup Summary Table

	Units	Receiver 1
Inlet flow	kmol/hr	0.067973
Total holdup	kmol	0.042646
Holdup		
ETHANOL	kmol	0.027394
WATER	kmol	0.015252
Composition		
ETHANOL	kmol/kmol	0.642363
WATER	kmol/kmol	0.357637

Operating Step Results

Main
Operating Step Results Table

Step	Step End Time	Units
Reflux_40	0.16668	hr
Reflux_50	0.33335	hr
Reflux_60	0.500017	hr
Reflux_70	0.0	hr
Reflux_80	0.0	hr

Multiple Batch Results

Main
Batch number : 1
Number of batches : 1
Time from start of batch : 0.500017
Time from start of first batch : 0.500017 hr

Results

Results at: End of Operating Step : Reflux_70
Simulation time: 0.666689 hours
Batch Number: 1

Pot Results

Holdup

Liquid level : 0.077423 m

Total molar holdup : 0.407853 kmol

Total mass holdup : 7.61475 kg

Liquid volume : 0.008395 m3

Molar boilup rate : 0.166087 kmol/hr

Mass boilup rate : 3.9568 kg/hr

Heat transfer summary

Temperature : 96.7997 C

Jacket duty : 0.008592 GJ/hr

Coils duty : -0.001375 GJ/hr

External exchanger duty : 0.0 GJ/hr

Duty for heat transfer with environment : 0.0 GJ/hr

Net duty : 0.007217 GJ/hr

Jacket

Duty : 0.008592 GJ/hr

Heat transfer area : 0.666387 m2

Heat transfer area in contact with liquid : 0.205754 m2

Medium temperature : 120 C

Coils

Heat exchanger

Profile Results

TPFQ

Basis : Mole

TPFQ Table

Stage	Temperature	Pressure	Duty	Liquid flow	Vapor flow
	C	bar	GJ/hr	kmol/hr	kmol/hr
Drum	78.8999	1.01325	0.0	0.171316	0.0
Condenser	79.0842	1.01325	-0.006755	0.119921	0.0
2	80.807	1.02754	0.0	0.121944	0.171317
3	84.0631	1.04182	0.0	0.126005	0.17334
4	89.2109	1.05611	0.0	0.127079	0.177401
5	93.855	1.07039	0.0	0.120503	0.178475
6	95.8263	1.08468	0.0	0.115664	0.171899
7	96.4272	1.09896	0.0	0.114691	0.16706
Pot	96.7997	1.11325	0.007217	0.0	0.166087

Composition

Basis : Mole

Liquid Composition Table

Stage	ETHANOL	WATER
Drum	0.616052	0.383948
Condenser	0.616051	0.383949
2	0.460245	0.539755
3	0.274688	0.725312
4	0.118022	0.881978
5	0.044203	0.955798
6	0.026142	0.973858
7	0.023781	0.976219
Pot	0.02335	0.97665

Vapor Composition Table

Stage	ETHANOL	WATER
Drum	0.0	0.0
Condenser	0.701919	0.298081
2	0.616051	0.383949
3	0.506454	0.493546

4	0.373626	0.626374
5	0.261544	0.738456
6	0.215228	0.784772
7	0.207609	0.792391
Pot	0.207043	0.792957

Condenser Results

Main
Inlet temperature : 80.807 C
Outlet temperature : 79.0842 C
Outlet pressure : 1.01325 bar
Outlet vapor fraction : 0.0
Outlet liquid fraction : 1
Cooling
Basis : Mole
Duty : -0.006755 GJ/hr
Condensing duty : -0.006755 GJ/hr
Liquid flow rate : 0.171317 kmol/hr
Composition
Basis : Mole
Composition Table

	ETHANOL	WATER
Inlet composition	0.616051	0.383949
Liquid composition	0.616051	0.383949
Vapor composition	0.701919	0.298081

Reflux Results

Reflux
Basis : Mole
Reflux flow rate : 0.119921 kmol/hr
Reflux ratio : 2.33333
Drum Holdup
Basis : Mole
Liquid level : 0.5 m
Liquid holdup : 2.16872e-08 kmol
Liquid volume : 1e-09 m3
Temperature : 78.8999 C
Heat transfer to environment : 0.0 GJ/hr
Drum Composition
Basis : Mole
Composition Table

	Units	ETHANOL	WATER
Component holdup	kmol	1.33585e-08	8.32864e-09
Liquid composition		0.616052	0.383948

Distillate Results

Main
Basis : Mole
Receiver taking liquid : 1
Liquid flow rate : 0.051395 kmol/hr
Composition
Basis : Mole
Composition Table

	ETHANOL	WATER
Liquid composition	0.616052	0.383948

Holdup Summary Results

Column

Column Table

	Units	Total	Pot Liquid	Tray/Packing Liquid	Reflux Drum
Total	kmol	0.407853	0.407853	2.38111e-07	2.16872e-08
Holdups					
ETHANOL	kmol	0.009523	0.009523	2.90961e-08	1.33585e-08
WATER	kmol	0.39833	0.398329	2.09015e-07	8.32864e-09
Composition					
ETHANOL	kmol/kmol	0.02335	0.02335	0.00291	0.616052
WATER	kmol/kmol	0.97665	0.97665	0.020902	0.383948

Distillate

Distillate Holdup Summary Table

	Units	Receiver 1
Inlet flow	kmol/hr	0.051395
Total holdup	kmol	0.051277
Holdup		
ETHANOL	kmol	0.033295
WATER	kmol	0.017982
Composition		
ETHANOL	kmol/kmol	0.649324
WATER	kmol/kmol	0.350676

Operating Step Results

Main

Operating Step Results Table

Step	Step End Time	Units
Reflux_40	0.16668	hr
Reflux_50	0.33335	hr
Reflux_60	0.500017	hr
Reflux_70	0.666689	hr
Reflux_80	0.0	hr

Multiple Batch Results

Main

Batch number : 1
Number of batches : 1
Time from start of batch : 0.666689
Time from start of first batch : 0.666689 hr

Results

Results at: End of Run
Simulation time: 0.833359 hours
Batch Number: 1
Results reported for last step